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**ECBC-SP-010**

**DESTRUCTION OF CHEMICAL WEAPONS:  
EVALUATION OF THE  
DONOVAN CONTAINED DETONATION CHAMBER (CDC)  
POELKAPELLE, BELGIUM**



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**July 2002**

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**Aberdeen Proving Ground, MD 21010-5424**

**20021015 089**

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<b>REPORT DOCUMENTATION PAGE</b>			<i>Form Approved</i> <b>OMB No. 0704-0188</b>	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave Blank)		2. REPORT DATE 2002 July		3. REPORT TYPE AND DATES COVERED Final; 01 May – 01 Jul
4. TITLE AND SUBTITLE Destruction of Chemical Weapons: Evaluation of the Donovan Contained Detonation Chamber (CDC), Poelkapelle, Belgium			5. FUNDING NUMBERS  NONE	
6. AUTHOR(S) DeBisschop, Herbert C. (RMA), and Blades, Timothy A. (ECBC)				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) RMA, Department of Chemistry (CHCH – Blok H), Hobbemastraat 8, 1000 Brussels, Belgium DIR, ECBC, ATTN: AMSSB-RCB-C, APG, MD 21010-5424			8. PERFORMING ORGANIZATION REPORT NUMBER  ECBC-SP-010	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES POC: John Schwarz, ATTN: AMSSB-RCB-CM, 410-436-2150				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words)  The Royal Military Academy (RMA) of Belgium was requested by the Belgium Minister of Defense to study alternatives to destroy WWI chemical munitions in an environmentally safe manner (RMA Study F0016). The RMA arranged for DeMil International to ship one of its transportable Contained Detonation Chamber systems to Poelkapelle so that the RMA could evaluate its potential to destroy these munitions. The U.S. Army Corp of Engineers (ACE), (Huntsville, AL) Division and U.S. Army Edgewood Chemical Biological Center provided technical assistance in this evaluation. The testing was performed from May - June 2001. Shells containing DA, HD, and CG were tested. Air monitoring technologies, including MINICAMS, the GC/MS equipped with a DYNATHERM Thermal Desorption Unit to analyze DAAMS, and the OPFTIR were used onsite during the experiment. Monitoring was applied at the exhaust vent, expansion chamber, detonation chamber, and the perimeter of the system. The CDC performed as designed to contain the blast and overpressure from the explosive treatment of chemical warfare agency (CWA) shells. Treating CWA shells using an oxygen-enriched "sheet" high explosive was demonstrated; however, residual levels of CWA were observed in the detonation chamber, expansion chamber, and the ductwork leading to/from the bag-house.				
14. SUBJECT TERMS Royal Military Academy Contained Detonation Chamber Destruction of chemical weapons			15. NUMBER OF PAGES  171	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT <b>UNCLASSIFIED</b>	18. SECURITY CLASSIFICATION OF THIS PAGE <b>UNCLASSIFIED</b>	19. SECURITY CLASSIFICATION OF ABSTRACT <b>UNCLASSIFIED</b>	20. LIMITATION OF ABSTRACT  <b>UL</b>	

# **DESTRUCTION OF CHEMICAL WEAPONS: EVALUATION OF THE DONOVAN CONTAINED DETONATION CHAMBER (CDC) POELKAPELLE, BELGIUM**

## **1. EXECUTIVE SUMMARY**

During World War I, a large number of chemical weapons were used in a concentrated area within Belgium. A considerable percentage of those weapons were duds. Several hundred chemical munitions are retrieved yearly in Belgium mainly in an area of roughly 600 square kilometers.

The destruction of these old chemical weapons is complicated by the degree of corrosion, the apparent stability of the agents, the imminent danger for intoxication, and a possibility of accidental explosion.

In October 1999, a dismantling facility was started in Poelkapelle, Belgium to separate explosives and chemical agents. The explosives were detonated and the agents were incinerated off-site. CLARK munitions, containing diphenylchloroarsine or diphenylcyanoarsine, could not be dismantled by this process because of the difficulty separating the explosives and chemical agents, resulting in an inability for disposal in industrial waste incineration plants.

The Royal Military Academy of Belgium (RMA) was requested by the Belgium Minister of Defense to study alternatives to destroy these WWI chemical munitions in an environmentally safe manner (RMA Study F0016). RMA arranged for DeMil International to ship one of its transportable Contained Detonation Chamber systems (Model T-10 equipped with a bag house for particulate emissions control) to Poelkapelle so that the RMA could evaluate its potential to destroy these munitions. A CDC for conventional munitions is composed of a detonation chamber (with pea gravel to attenuate the detonation effect), an expansion chamber and an air pollution control unit (APCU), consisting of six particle filters and a fan. For testing purposes, the CDC was modified to reduce toxic emissions by installation of a carbon/HEPA filtration system. The general aim of this testing was to evaluate the capacity of the CDC to completely eliminate any explosives and to destroy a large part of the chemical agents; any remaining material was then collected as toxic waste to be incinerated off-site.

The U.S. Army Corps of Engineers (USACE) Huntsville (AL) Division and the Edgewood Chemical Biological Center (ECBC) provided technical assistance in this evaluation. The focus of this support was collecting and analyzing air samples from the detonation plumes as a supplement for the RMA effort in this area.

The testing was done in two phases. Phase I tests, which were conducted from May 14-23, 2001, involved a robust evaluation of the capability of the DeMil CDC systems to destroy three types of WWI munitions currently stored at Poelkapelle. Phase I

tests consisted of taking samples of the detonation plume at three points on the system; the expansion chamber, the duct between the bag house and the carbon/HEPA filters, and pea gravel and wipe samples in the detonation chamber. During Phase II (June 14-July 13, 2001), a similar approach was followed, but sampling emphasis was concentrated on personnel monitoring and monitoring at the carbon/HEPA filters exhaust.

ECBC focused its analytical effort on Mustard (HD), Chloropicrin (PS) and Phosgene (CG). Significant amounts of the mentioned agents were detected in the expansion chamber and the exhaust duct leading to the carbon/HEPA filtration system. HD was not detected at the perimeter monitoring positions and the exhaust vent, but was found present at the door exhaust and sample pumps worn by protected operators. The Open-Path Fourier Transformer Infrared Spectrometer (OPFTIR) was used above the detonation chamber door during Phase I. The instrument did not detect any agent during the HD, PS/CG, and CLARK detonations. During Phase II the instrument was positioned after the exhaust vent. The OPFTIR did not detect any breakthrough. The OPFTIR observed increased levels of carbon monoxide, methane, ethylene, and acetylene corresponding to the detonations.

Dust particles were effectively retained by the APCU bag house filtration system. Chloride analyzed on quartz particle filters show that the ratios of volatile chloride to non-volatile chloride were large for the CG/PS munitions, somewhat smaller for the HD/CB munitions and very small for the DA munitions. Solid (non-volatile) chloride was either not found or found in low concentrations in the duct downstream of the bag house. The non-volatile chloride concentrations for the three types of munitions were similar.

Residues on the wall of the detonation chamber and in the pea gravel were representative of the non-volatile agents HD and DA and less so or not detected for the more volatile compounds. Airborne concentrations in expansion chamber and duct are higher for Chlorobenzene, and much less for the other CWA.

It was occasionally noticed that very low CWA concentrations, relating to the agent contents of the shells destroyed were measured on TENAX tubes at the exhaust vent. This indicated failure of the HEPA/Active Carbon filtering system. However, neither the OPFTIR system nor the perimeter monitoring system detected any emissions.

Personnel monitoring with solid sorbent tubes was applied systematically during the second phase of testing. The highest exposure levels were noticed to protected personnel directly involved with the operation of the CDC and when decontaminating the detonation chamber.

The mass of the undestroyed agent was estimated by combining results from the sampling at detonation chamber, expansion chamber (including the solid soot/ash deposit), the bag house filters, the exhaust duct and the exhaust vent. It appears that destruction of CG, PS and HD was good, but DA and Chlorobenzene were not as effectively destroyed.

The results of the tests indicate that the CDC performed as designed to contain the blast and the overpressure from the explosive treatment of the shells. Additionally, the personnel protective equipment worn by explosive technicians was adequate in protecting them from exposure to chemical agents.

## 2. INTRODUCTION

### 2.1 The Use of Chemical Warfare Agents in World War I

It is generally accepted that the first large scale use of chemical weapons began on the Western front on 22 April 1915, when specialized German troops opened some 6000 cylinders (the equivalent of about 160 tons) of chlorine along a front line of nearly 7 kilometers north of Ypres, Belgium. British troops retaliated in a similar manner in September 1915.

Prior to April 22, gas shells filled with irritating agents (xylylbromide, benzyl bromide and bromoacetone) were used, however, with limited success. It was soon realized that in order to be effective, a sufficiently high concentration of CWA had to be generated over a sufficiently long time. Cylinder attacks were capable of this, but they were strongly dependent upon meteorological conditions, more particular wind speed and direction. This method of chemical warfare disappeared in 1916 and was replaced by rapid firing howitzers (Germany and France), while the British developed the Livens projector.

The search for novel agents was rather chaotic and lacked a systematic approach. In 1916, mainly non-persistent lethal agents such as Phosgene, Diphosgene and hydrogen cyanide were used in combination with irritants (Chloropicrin and arsenic trichloride). In the summer of 1917, the Germans introduced Mustard gas (Yperite, LOST). This agent color-coded Gelbkreuz (Yellow Cross) was used in combination with (di)phosgene, Grünkreuz (Green Cross) and a sternutatory agent, diphenyl chloroarsine (CLARK), color coded Blaukreuz (Blue Cross). It took the British and French until 1918 before they had mastered the industrial scale synthesis of Mustard.

In total, some 60 agents or combinations of agents were used. The physical dimensions and the chemical contents of the majority of munitions discovered in Belgium are given in Tables 1 and 2. The physical properties of the relevant chemical agents, additives and explosives are listed in Table 3. Table 4 lists the fill characteristics of 77 mm LFKGr shells. This shell type was used exclusively for the testing at Poelkapelle.

### 2.2 Particularities of Use of CWA in WW I

From October 1914 till September 1918, fighting from the Flemish coast to the Swiss border, known as the front zone moved only a few kilometers despite frequent attempts to breakthrough. Heavy shelling could last for days preceding major attacks. Non-persistent chemicals were mainly used immediately before the attack. For area denial or harassing, more persistent chemicals were employed.

Shell production standards of those days cannot be compared with present munitions production standards. Moreover, the German factories were in short supply of basic ingredients for the fuse production and had to rely upon *ersatz* (substitutes). Hence, the number of duds increased significantly. It was estimated in the last year of the war about one out of three German shells fired did not explode. Combined with the high number of shells produced (Prentiss<sup>1</sup> estimates the total number of shells produced in WWI at about 1.5 billion, roughly 4.5% of which was chemical. All shells were used on a

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<sup>1</sup> A.M. Prentiss, Chemicals in War, McGraw-Hill, p. 658 (1937)

rather small area. This accounts for the large quantities of unexploded munitions that are still found in the former front zone area.

### **2.3 Treating Old Chemical Weapons in Belgium before 1998<sup>2</sup>**

Immediately after WWI, civilians began searching the former front zone to recover valuable metals such as copper from the driving band of artillery shells. Later small companies, such as Pickett and Son were set up to treat munitions at a larger scale. An official report of 15 September 1922 mentioned the destruction of twenty eight thousand tons of munitions, roughly one fourth of them being chemical. In 1920, an organization named SDM (Service for Destruction of Munitions) was created. Its principle task was to retrieve and destroy munitions. Another division of the Belgian Army, the Shore Service was reported to have destroyed some fifteen hundred tons of old munitions, six percent of which were chemical. Other data on destruction was almost non-existent. Another way of disposal was sea dumping. Immediately after the war, the British Expeditionary Corps dumped large amounts of munitions and probably other warfare material in an off the coast area near Knokke, on a sand bank known as "Paardenmarkt" (Horse Market).

No reports were available on the ways of disposal used in the period from the early 1920's till after the World War II. The Belgian Navy dumped 810 tons of chemical munitions in the Gulf of Biscay from 1954 till 1972. Munitions were first put in metal shell casings that were encased in concrete.

In 1972, as a consequence of Belgium signing the Oslo Convention on the Prevention of Sea Dumping, the practice of sea dumping was stopped. However in 1980, the Belgian Government invoked the emergency provision of the Convention and obtained a license for the dumping of some 225 tons of supposedly chemical munitions. The operation was successfully carried out in October 1980.

The Bomb Disposal Unit (DOVO) of the Belgian Armed Forces yearly retrieved some 200 tons of old munitions from 1980 to 1998. As a rule there was no systematic search for old items unless indications existed that a larger storage may be uncovered. Most items were found incidentally by road workers, construction workers, and by farmers working in the fields. Retrieved munitions were collected at the DOVO barracks in Poelkapelle where they were cleaned and tentatively identified. About 90 percent of the munitions were identified as conventional. They were destroyed by covered-earth detonation. The remaining munitions were stored outside on pallets under roofing awaiting the development of appropriate methods for disposal.

The Chemical Weapons Convention (CWC) entered into force in 1997. Under this Convention the WWI chemical munitions retrieved in Belgium were declared as Old Chemical Weapons and were to be destroyed as chemical waste.

### **2.4 Dismantling Old Chemical Weapons**

In the early eighties the question of how to deal with old chemical weapons in an environmentally sound manner became an urgent matter in Belgium. However, it took until 1991 before an important study contract was awarded to the Belgium-based firm

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<sup>2</sup> For a detailed overview see J.P. Zanders, The Destruction of Old Chemical munitions in Belgium, in SIPRI Chemical & Biological Warfare Studies, Volume 16: "The Challenge of Old Chemical Munitions and Toxic Armament Wastes", T. Stock and Kh. Lohs, Eds., Oxford University Press, 1997.

COPPEE-COURTOY, who continued from initial studies by the Technical Division of the Army and by a firm named SGS-DEPAUW and STOKOE<sup>3</sup>. A building contract was awarded in November 1992 and construction of a dismantlement facility started in June 1993 and ended in December 1994. However, it took until December 1997 before the X-ray Identification System was allowed to operate and until October 1999 before the Dismantlement Facility was routinely working. This was due mainly to the difficulties in establishing acceptable safety procedures.

For the majority of old items retrieved, external characteristics allow for a positive identification as non-chemical. Doubtful cases are X-rayed. Since the internal geometry of the chemical shells is fundamentally different from conventional ones, this analysis allows for a positive identification as chemical. However, the actual chemical contents of the shell cannot be stated with certainty unless other non-destructive testing tools, such as Neutron Activation Analysis is used.

## **2.5 The Problem of CLARK-type Munitions**

CLARK I (diphenylchloroarsine) was introduced by the German Forces in July 1917. Diphenylchloroarsine is a solid or syrup like compound, when impure and was intended as a "mask breaker". The compound was pulverized and dispersed as an aerosol through the mechanical and heat effects of the explosion. The microscopic particles could easily penetrate the rudimentary gas masks in use. CLARK II (diphenylcyanoarsine), a similar compound, is more toxic and more effective at lower doses. Only Germany used these agents. They were delivered in artillery shells. The agent was contained in a glass bottle and surrounded by an explosive charge to ensure adequate dispersion. (photograph 4.9)

CLARK-type munitions are easily identified by X-ray analysis due to the presence of a glass bottle. The munitions are dismantled, first by cutting the head and base off, and then pushing the contents out of the cylinder with the aid of a piston. The waste produced was a mixture of CLARK, glass, and explosive that is not accepted by the industrial waste incinerating company for safety reasons. Essentially two possibilities remain; either finding ways to adequately destroy the CLARK-explosive mixture, or to look for alternative ways to destroy CLARK-type shells.

## **2.6 Other Reasons for Searching New Technologies**

Dismantling CLARK munitions requires three operations (two milling and one press), compared to other shells, which only need one or two operations. This operation was time-consuming and considerably reduced the output of the Dismantling Facility. Furthermore, eighteen years passed from the time sea dumping was banned until the startup of the facility. As a result, a stock of more than 10,000 chemical shells was built up. A need for an increased dismantlement/destruction pace was evident. In addition, the degree of corrosion of the old shells was ever increasing and resulted in the need to minimize handling.

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<sup>3</sup> DEPAUW and STOKOE, an affiliate of Société Générale de Surveillance (SGS) dealing with control and measurement in the area of industrial safety.



## **2.7 The Contained Detonation Chamber – a Possible Alternative for Dismantling**

The Royal Military Academy of Belgium (RMA) was requested by the Minister of Defense to study alternatives to destroy WWI chemical munitions in an environmentally safe manner (RMA Study F0016). Emissions testing of the DeMil International Contained Detonation Chamber (CDC) system being used to destroy munitions containing conventional explosives<sup>4,5</sup> indicated that these systems, if combined with an emissions control system, might be a suitable disposal technology. RMA arranged for DeMil International to ship one of its transportable CDC systems (Model T-10 equipped with a bag house for emissions control) to Poelkapelle. RMA planned to evaluate its potential to destroy these munitions. RMA asked the U.S. Army Corp of Engineers (USACE) Huntsville (AL) Division and the Edgewood Chemical Biological Center (ECBC) to provide technical assistance in this evaluation. The primary focus of this support was in collecting and analyzing samples from the detonation plumes as a supplement for the RMA effort in this area. The testing was done in two phases. Phase I tests were conducted from 14-23 May, 2001 involved a robust evaluation on the capability of the DeMil CDC systems to destroy three types of WWI munitions currently stored at Poelkapelle. The Phase I tests mainly consisted of taking samples of the detonation plume from three parts of the system; the expansion chamber, the duct between the bag house and the carbon/HEPA filters, and pea gravel and wipe samples in the detonation chamber. A similar approach was followed during Phase II, but emphasis was put on personnel monitoring and monitoring at the carbon/HEPA filters exhaust.

## **3. DESCRIPTION OF THE DEMIL INTERNATIONAL T-10 SYSTEM**

In 1999, DeMil International, Huntsville, AL developed a transportable version of its CDC technology for use in destroying unexploded ordnance (UXO). In January 2000, the Department of Defense Explosive Safety Board approved DeMil's Model T-10 CDC system for destroying ordnance containing up to the equivalent of 5.9 kg of TNT. In May 2000, the USACE began using a Model T-10 for destroying 81 mm mortars at the Massachusetts Military Reservation (MMR). In January 2001, emissions testing conducted by a USACE contractor with oversight from the U.S. Environmental Protection Agency (USEPA) showed that the T-10 emissions were well below levels that would endanger human health and the environment.<sup>6</sup>

Although the T-10 system that was used at Poelkapelle was modified to make it suitable for destroying munitions containing chemical agents, it was still very similar in

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<sup>4</sup> Emissions Test Report, For the T-10 Transportable Donovan Contained Detonation Chamber at Massachusetts Military Reservation (MMR). Prepared for DeMil International, Inc. By Shield Environmental Associates, Inc. 2456 Fortune Drive, Suite 100, Lexington, KY 40509, February 2001.

<sup>5</sup> BGAD (Blue Grass Army Depot) Detonation Chamber Data Collection Test Report, Phases I and II. Prepared for DeMil International, Inc. by El Dorado Engineering, Inc. 2964 West 4700 South, Suite 109, Salt Lake City, UT 84118, August 2000.

<sup>6</sup> Emissions Test Report, For the T-10 Transportable Donovan Contained Detonation Chamber at Massachusetts Military Reservation (MMR). Prepared for DeMil International, Inc. By Shield Environmental Associates, Inc. 2456 Fortune Drive, Suite 100, Lexington, KY 40509, February 2001.

design to the T-10 operated at the MMR. The procedures used to prepare the munitions for destruction were similar to those used at the MMR.

### **3.1 Description and Function of the Components of the T-10, Modified for the Poelkapelle Testing (Photograph 4.6)**

#### **3.1.1 Detonation Chamber**

The detonation chamber was equipped with a double wall fabricated from A-36 grade steel plate. The exterior dimensions were 2.0 m wide, 2.0 m long, and 2.1 m high. The interior wall was lined with hardened, abrasive resistant armor plating and the space between the walls was filled with dry silica sand. The weight of the chamber with silica sand was approximately 18,400 kg. During operation, the floor of the chamber is covered with 12 cm (0.35 m<sup>3</sup>, 500 kg) of pea gravel and thin-walled plastic bags containing water are suspended in the detonation chamber. Typically a 1.5 to 1.0 ratio of water to total energetic is used. This was subject to change based on the type of energetic munitions and other unique conditions. The pea gravel and water were used for two reasons. First, they attenuated the shockwave, over-pressure, and hot gases released by the detonation, thereby protecting the integrity of the chamber. Second, they released wet dust particles and water droplets which may have served as nuclei for the detonation products to adhere to, which aids in the collection of the emission products by the APCU.

#### **3.1.2 Expansion Chamber**

The gases and particles released by the detonation vent into the single walled steel expansion chamber which has interior dimensions of 2.3 m x 2.0 m x 2.0 m. This chamber is reinforced with channel steel for strength and weighs 2,400 kg. It both attenuated the over-pressure and heat remaining from the detonation and aided in the removal of particles and condensable materials from the detonation gas stream.

#### **3.1.3 Air Pollution Control Unit (APCU)**

The APCU was a Model TD-573 bag house that was designed by TORIT Industries specifically for the T-10. It is 3.1 m high, 2.5 m wide, and 2.1 m long. It had a 0.3 m<sup>3</sup> hopper and a constant speed blower, which operated at 73 m<sup>3</sup> per minute. The particulates from the detonation were cleared from the filter by automatic reverse airflow. Electrical requirements were 480 volts (3 phase) at 20 amps.

### **3.2 Modifications**

#### **3.2.1 Sampling and Secondary Door (Photographs 4.5 and 4.7)**

Six ports were added to the expansion chamber for sampling procedures. Three ports were grouped at the mid-point of the expansion chamber. The other 3 ports were above the inspection port. The ports were spaced in a 30.5 cm cluster. The interior diameter size of each port was 1.9 cm.

With the fielding of several T-10 Contained Detonation chamber systems in the U.S. and in Europe, DeMil had developed a modification of the access door system designed to eliminate the fugitive emissions that had been noticed to occur during

detonation. This redesign consisted of the fabrication of a secondary door that closed and sealed over the access door opening on the detonation chamber. This secondary door formed an airtight seal over the access door. The secondary door was connected to the Air Pollution Control Unit (APCU) via a vinyl pipe that provided negative pressure between the access door and the secondary door. Any fugitive emissions from the access door were captured by the vacuum behind the secondary door and vented to the APCU for filtering.

### **3.2.2 HEPA/Active Charcoal Filter (photograph 4.2)**

A duct that leads to a set of four ANDAIR AG, Model GF 1200 E/S Activated Charcoal/HEPA Filter units (in parallel arrangement) was attached to the exhaust stack of the T-10 after the bag house filter. The blower attached to this system operated at a flow rate of 80 m<sup>3</sup> per minute, slightly higher than the APCU flow rate. All emission gases from the T-10 passed through these Carbon/HEPA filter units before they entered the environment.

## **3.3 Principles of Operation**

- Standard Operating Procedures<sup>7</sup> were applied.
- Additional safety measures were applied for handling CWA munitions.

### **3.3.1 Preparation of the munitions**

- Munitions were wrapped in PBX<sup>8</sup> sheet explosive, also called DBS<sup>9</sup> and transported to the site (about 300 meters). Quantities of explosive used per shell are listed in Table 8.
- Operators wore standard NBC protective gear with gasmask.

### **3.3.2 Preparation of the detonation**

- Water and additives were hung in the detonation chamber.
- The munition was placed in the detonation chamber and a detonator was fixed (photograph 4.8).
- Doors were closed and the munition was detonated.
- Throughout the loading of the chamber until after the closing of the doors operators wore NBC protective clothing and a pressurized<sup>10</sup> gas mask.

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<sup>7</sup> Standard Operating Procedure for Destroying Unexploded Ordnance and Maintenance and Inspection for the T-10 Transportable Contained Detonation Chamber "Donovan Blast Chamber". Prepared by DeMil International, Inc., 221 East Side Square, Huntsville, AL 35801. September 2001.

<sup>8</sup> PBX Sheet, Aluminum Sheet, Manufactured by Donovan Commercial Industries, P.O. Box 909, 400 Williams Mine Road, Nortonville, KY 42442

<sup>9</sup> "Donovan Blast Sheet"

<sup>10</sup> For Phase I, a normal gas mask with HEPA/Active Charcoal Filter was used. During the second phase CDC operators were equipped with supplied air apparatus.

## **4. TEST OBJECTIVES AND METHODOLOGY**

The test objective was to determine the effectiveness of the Contained Detonation Chamber (CDC) for containing and destroying chemical agent filled munitions.

### **4.1 Basic Philosophy**

The CDC was operated in a similar fashion to the dismantlement facility currently operated at Poelkapelle. Containment was essential for the operation of both processes. Exposure of the operators was monitored and had to stay under well-established threshold values. After dismantlement, toxic contents and residual waste were double packed and incinerated at an off-site industrial toxic waste kiln.

It was expected that explosives and a large part of the toxic contents of the chemical shells were to be destroyed in the CDC. This was done by the combination of the detonation and the subsequent fireball created by the explosive sheets wrapped around the shell. Residues of toxic compounds in the detonation chamber, expansion chamber and filters were packed as toxic waste and later incinerated. It was mandated that during the whole operation, exposure of operators and emission to the environment were to stay under set limits.

### **4.2 Selection of Munitions to be Destroyed**

Tables 1 and 2 provide information on the size and chemical fill of the vast majority of WWI munitions stored at Poelkapelle. Table 3 provides information on selected physical properties of each of the chemicals contained in these munitions. The chamber was designed to test the destruction of 105 mm shells. However, for safety reasons it was decided not to destroy rounds that exceeded the 77 mm caliber. The final choice of the munitions types was a joint decision; from the Belgian side priority was put on DA (CLARK) type shells, while the US was more interested in testing the destruction of shells containing Mustard. Details of the chemical fill of these shells are given in Table 4. All shells selected were of the type LFKGr.

### **4.3 Approaches by Phases**

During Phase I, a limited number of shells containing DA, Mustard and Phosgene were tested. The objectives of this first phase were to make an initial evaluation of the possibilities offered by the CDC T-10 for the destruction of HD, CG and DA, to estimate destruction efficiencies (if possible), and to establish additional parameters to be monitored during Phase II.

It was concluded from Phase One that the contained detonation chamber was capable of effectively destroying the explosive contents of the shells and to a lesser extent its chemical contents. Explosive residues were not detectable or were just above the limit of detection. Airborne residues of the toxic fill were quantified in the expansion chamber and after the bag house. Values ranged from a few  $\text{mg/m}^3$  to a few tens of  $\text{mg/m}^3$ , except for Phosgene and Chloropicrin, where in one case a value of several hundreds of  $\text{mg/m}^3$  was found.

Residues in pea gravel typically showed residual contaminations more than 100  $\text{mg/kg}$  gravel. As shown in Tables 1 and 2, it was not possible to know the exact mass of explosive or CWA in a specific WWI munition. It was not possible to determine absolute

destruction efficiency from the planned detonation experiments because of these uncertainties in the masses, and the difficulty in sampling the interior parts of the T-10 system.

On this basis, the following objectives were set for Test Phase II:

- To evaluate workers safety through monitoring for exposure.
- To optimize destruction efficiency using additional explosives, additives, and cleansing detonations while systematically sampling the detonation chamber.
- To quantitatively evaluate the residual toxic waste.

#### **4.4 Analyses and Monitoring Philosophy (Appendix 1 - Figure 1.3.1.8.1 Schematic of Monitoring around CDC)**

Choices for monitoring positions were limited by time and equipment. Due to the lack of a vapor containment system (VCS), the perimeter around the Detonation Chamber was chosen for documentation of exposure to the environment and personnel. The chamber was equipped with a particulate and carbon filter system. Sampling positions were setup at these positions to monitor for possible filter breakthrough. Samples were collected at the detonation chamber and the expansion chamber to determine the efficiency of the detonation. Monitoring was also provided at the detonation chamber door to determine if the possibility for migration exists. In addition, operation personnel were monitored for contamination. Swab and pea gravel samples were collected between munition detonations to test for contamination to the walls of the detonation chamber.

## **5. CONDENSED METHODS AND SAMPLING (APPENDIXES 1 AND 2)**

### **5.1 Plume samples**

The T-10 detonation process was designed to attenuate the blast effect and to cool the detonation plume very quickly. Based on the quantities of water, explosive and chemical agents used in the Poelkapelle tests, the temperature of the detonation plume as it enters the expansion chamber was expected to be less than 60 °C. The volume of the plume was expected to be less than 2.5 m<sup>3</sup> because of the relative positions of the baffles in the expansion chamber. The detonation plume was expected to displace a volume of the air from the expansion chamber equal to its own volume without mixing with it (plug flow). Furthermore, since the combined volume of the detonation chamber and expansion chamber was much larger than the detonation plume (24 m<sup>3</sup> versus 2 to 5.4 m<sup>3</sup>), the detonation plume was expected to remain within the bag house and expansion chambers. At the time of the detonation, the blowers on the bag house and charcoal/HEPA filter systems were operating, but because the door to the detonation chamber was closed the air was flowing in the system.

The small volume of the detonation chamber/expansion chamber (24 m<sup>3</sup>) compared to the flow rate of the blowers (73 m<sup>3</sup>/min) and the batch operation of the T-10 process, made it impractical to collect representative samples from the plume while the chamber was being evacuated. For these reasons, it was decided to collect the samples while the air in the T-10 chambers and the Air Pollution Control System (Bag house,

charcoal-HEPA filter) was not flowing (quiescent). However, it was anticipated that these samples collected as described in generic terms, hereafter, were representative of the plume produced when the T-10 was operated in the normal fashion.

## **5.2 Residue samples**

At the end of a shot sequence, wipes were taken from the inside of the chamber (100m<sup>2</sup>) using pre-wetted cellulose kept in closed glass containers until use, as described in Appendix 1.3.1.5. Pea gravel samples were taken from the inside of the detonation chamber and collected in glass bottles (Appendix 1.3.1.7). After the end of each phase and at set times during Phase II, samples were taken in the expansion chamber (soot/dust) and from the aerosol filters (bag house filters) when they were replaced.

## **5.3 Monitoring Samples**

- The perimeter of CDC was monitored for HD with DAAMS.
- Personnel samples: Operators of CDC were monitored using personal air sampling pumps with TENAX/DAAMS tubes.
- During Phase II, the exhaust vent after the Charcoal Filter was systematically monitored with DAAMS, TENAX, and OPFTIR.

## **5.4 Short Description of the Sampling and Methods**

Tables 5 and 6 contain a description of the methods that were used to collect and analyze the detonation plume samples during the Phase I and Phase II, respectively. Table 7 indicates typical sampling flows and times. The reasons each method was selected is provided below along with a description of each sampling and analysis procedure. Details of sampling procedures are given in Appendix 1.

ECBC applied three types of air monitoring technologies onsite during the experiment. These included Miniature Chemical Air Monitoring System (MINICAMS), the Gas Chromatograph/ Mass Spectrometer (GC/MS) equipped with a DYNATHERM Thermal Desorption Unit to analyze DAAMS (Depot Area Air Monitoring System), and the Open Path Fourier Transform Infrared Spectrometer (OPFTIR). Detailed methods are explained in Appendix 2.

RMA used air monitoring via a personal sampling pump and re-usable TENAX adsorbent tubes analyzed by Thermal Desorption and GC/MS. This type of analysis was a basic technique used for monitoring the past dismantlement facility. This method was supplemented by the use of charcoal adsorbents, ORBO32 for volatile organic compounds and ORBO24 for Phosgene. The air samples taken via bubblers and quartz filters were analyzed by Capillary electrophoresis via bubblers. Airborne particulate matter sampled by Millipore filters were analyzed for residual metals (Al, As, Cu and Pb) by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). Wipes and pea gravel were analyzed for residual CWA via extraction and GC/MS and for residual metals by ICP-AES. Where appropriate, soot samples from the expansion chamber and paper samples from the particulate filters in the bag house were analyzed by GC-MS and ICP-AES.

Detailed accounts of the methods are given in Appendix 2.

## **6. RESULTS (Appendix 5-9)**

### **6.1 DAAMS Analyzed by GC/MS**

DAAMS were collected and analyzed by GC/MS. Background samples were collected during high explosive detonations to determine a baseline for sampling. QP samples were collected daily throughout the entire project reinforcing the detection capabilities of the method.

During Phase I, HD was detected in the expansion chamber. HD was not detected at any other DAAMS positions, but HD degradation products and Chlorobenzene were detected in the exhaust duct. At the end of Phase I the chamber was decontaminated and the area was monitored. The only detection of HD during decontamination was found in the particulate filters that were sampled in a plastic bag that was heated in the sunlight for four hours. Prior to beginning Phase II of the project sampling setup was changed at the exhaust duct, door exhaust, and the expansion chamber. Due to the lack of equipment during Phase I, TYGON tubing was used as sampling apparatus, but in Phase II it was changed to TEFLON tubing. TEFLON was a better sampling medium than TYGON because it was non-reactive, containing less active sites to adsorb CWA. This change in sampling technique proved to be critical in Phase II, due to significant differences in results.

During Phase II of the project, sampling positions were added at the exhaust vent and for personnel monitoring. Background samples were also taken during high explosive detonations at the beginning of Phase II. HD was detected in the exhaust duct reinforcing the error in sampling technique during Phase I and also because the duct was not decontaminated. Significant HD results were found in the expansion chamber during Phase II. Samples were so saturated that they were unable to be analyzed due to the sensitivity of the instrument. Sample splits were also attempted unsuccessfully to minimize instrument contamination. HD was also detected with significance at the exhaust duct before the carbon filter system and the door exhaust. Additionally, HD was detected on samples worn by personnel operating detonation chamber. HD was not detected at the perimeter positions and exhaust vent.

The instrument detection limit for HD was 0.9 ng/tube. Positive results for HD were stated with minimum confidence level due to instrument saturation, unavailability of concentrated standards, and time. All results were calculated assuming HD response was linear.

### **6.2 MINICAMS**

MINICAMS results for Phase I and Phase II found significant amounts of Phosgene/Chloropicrin present in the expansion chamber and in the exhaust duct (between particulate and carbon filters). In Phase II of the project amounts of Chloropicrin were detected coming through the exhaust vent after the carbon filters. The method detection limit for PS was 0.17 mg/m<sup>3</sup> and for CG was 0.1 mg/m<sup>3</sup>. Concentrations detected well above Airborne Exposure Limit (AEL) are estimated due to instrument calibration levels.

Results for Mustard (HD) during both Phases showed significant detections of HD at the expansion chamber and the exhaust duct. HD was not detected in the

monitoring room or at the exhaust vent. The detection limit for HD was 0.00075 mg/m<sup>3</sup>. Positive results are estimated assuming HD response was linear.

### **6.3 OPFTIR**

The OPFTIR was used above the detonation chamber door during Phase I. The instrument did not detect any agent during the HD, PS/CG, and CLARK detonations. During Phase II the instrument was positioned after the exhaust vent. The OPFTIR did not detect any breakthrough. The detection limit for the instrument changed daily due path length, compounds being analyzed, and environmental factors. The OPFTIR observed increased levels of carbon monoxide, methane, ethylene, and acetylene corresponding to the detonations. The OPFTIR observed higher responses at the exhaust vent than positioned over the detonation chamber door.

### **6.4 Quartz Filters and Bubblers**

Quartz Filters and Bubblers were used only during the Phase I tests, served two purposes. The first purpose was to evaluate the effectiveness of the bag house in preventing the particles released by the detonation from reaching the charcoal/HEPA filters. The second purpose was to determine the concentrations of volatile and non-volatile chloride compounds in the EC and the duct. The effectiveness of the bag house in removing the dust and other solid/semi-solid materials present in the detonation plume was determined by comparing the particulate concentrations in the EC (upstream of the bag house) to that in the duct (downstream of the bag house). This was an important measurement because at least some of the explosive and CWA not destroyed by the detonation was expected to be adsorbed on the surface of the particles released from the pea gravel and the munitions casing by the detonation. The generation of this "dust" was a critical part of the emissions control system on the T-10. The particulate concentrations measured before (EC) and after (duct) the bag house are presented in Tables 10, 12 and 13. The results reported in these tables showed that the particulate concentrations in the duct were below the detection limit of the measurement system; even when concentrations of particulate measured in the EC were quite high. These results confirm the expectation that the bag house would quantitatively retain the particles released by the detonation. Similar results were found in the USA when conventional munitions were detonated at the Massachusetts Military Reservation (MMR) and the Blue Grass Army Depot.

A comparison of the chloride found on the filter (non-volatile chloride compounds) and the chloride found in the bubblers (volatile chloride, e.g., HCl) showed that the ratios of volatile chloride to non-volatile chloride were large for the CG/PS munitions, somewhat smaller for the HD/CB munitions and very small for the DA munitions (Tables 10, 12 and 13). Solid (non-volatile) chloride was either not found or found in low concentrations in the duct downstream of the bag house. The non-volatile chloride concentrations for the three types of munitions were quite similar.



## **6.5 Airborne Concentrations of CWM in the Expansion Chamber and Exhaust Duct (Tables 9,11,13-17,19, 23, 24, 27, 28)**

Airborne concentrations of volatile CWA in expansion chamber and duct, combined with results for pea gravel, gave a good indication of the destruction efficiency. Values were low (not detected to tens of  $\text{mg}/\text{m}^3$ ) for all CWA, except for Chlorobenzene where concentrations were in the range of grams per cubic meter.

## **6.6 Volatile Aromatic Hydrocarbons (VAC) in the Expansion Chamber and the Exhaust Duct (Tables 18, 21, 22, 25)**

The lowest values were associated with the detonation of CG/PS shells. There were no aromatics present in this agent mix (Table 18). For detonations of HD shells, the level of VAC increased significantly (Table 24). Still higher values were obtained from detonations of DA shells (Table 21). The original aromatic content of the shell is reflected in the VAC concentrations found in the expansion chamber.

## **6.7 Wipe Samples**

### **6.7.1 Phase I**

Some wipe samples were analyzed for explosive residues using a method routinely applied for forensic purposes at the Laboratory for Analytical Chemistry of RMA. Only RDX was occasionally detected and could be quantified for four detonations. Quantities of 28, 65, 92 and 630  $\mu\text{g}$  per wipe were found. Other wipe samples were analyzed for chloride where amounts of tens of  $\text{mg}$  per wipe were found.

### **6.7.2 Phase II**

During Phase II, wipe samples were taken systematically in duplicate at the upper right corner using a square frame of  $0.0225 \text{ m}^2$ . Samples were analyzed for CWA residues and for residual metals (Al, As, Cu and Pb). Since the sample was taken at the same place, it was assumed that contributions from former detonations could be neglected. Results are found in Tables 14-17, 19, 20, 23, and 24 under the column heading "wall DC".

Due to its high volatility, no residues of (di)phosgene were found on the wall of the Detonation Chamber. (Table 15, 16) As a rule, no residues of Diphosgene were found in any of the sample types. It was assumed that Diphosgene was converted to phosgene either in the fireball of the detonation<sup>11</sup> or upon long time storage possibly under the influence of the metal wall of the shell casing.

Residues of volatile liquids, such as Chlorobenzene and Chloropicrin were not detected or found in wall concentrations equivalent to tens of  $\text{mg}$  per  $\text{m}^3$  (Tables 14, 17, and 24). DA was found in concentrations higher than one gram per square meter. (Table 19).

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<sup>11</sup> S. Franke et al., Lehrbuch der Militärchemie, Militärverlag der DDR, Leipzig (1976), p.344.

### **6.8 Pea Gravel Samples**

During Phase II, pea gravel samples were taken in duplicate and following a systematic pattern. Results are found in Tables 14-20, 23-24, and 26 under the column heading "pea gravel".

It was obvious that residues of volatile agents in pea gravel were either low or not detected. This was augmented by the airflow that was generated when the door of the detonation chamber was opened. Detonation of shells containing the less-volatile Mustard or diphenylchloroarsine gave important values. The heterogeneous sampling matrix had an adverse affect on the precision of the results. Moreover, every sample was contaminated by carryover from previous detonations. It was necessary to take this into account when interpreting these values. Elemental concentrations of metals showed a continuous increase (accumulation).

### **6.9 Exhaust Monitoring**

Airborne concentrations measured after the main fan of the HEPA/Active Carbon filtering system are given in Table 27. It was occasionally noticed that non-negligible CWA concentrations were measured relating to the agent content of the shells destroyed. This indicated failure of the HEPA/Active Carbon filtering system and a measurable emission of CWA into the environment. Obviously, concentrations were rapidly diluted to non-detectable values since the perimeter monitoring (the relevant monitoring system was only at a few meters) shows a non detectable immission<sup>12</sup>. Moreover, the OPFTIR system did not detect any emission.

### **6.10 Monitoring of Potential Operator Exposure**

Table 28 showed the results of the personal monitoring. Monitoring was performed on protected personnel to approximate unprotected operators. It must be emphasized that monitoring pumps were switched off when the Detonation Chamber door was closed, thus resulting in maximum concentrations. As expected, detonating shells in rapid sequence and decontaminating/emptying the Detonation Chamber gave the highest levels of personal exposure (unprotected personnel).

### **6.11 Residues in Ashes and Soot in the Expansion Chamber**

At various decontamination/cleanup periods during Phase II samples were taken from the soot/ashes in the expansion chamber. Samples were taken systematically at the same spot and covering the same area. At the end of Phase II the expansion chamber was emptied. For 90 detonations, 108 kg of soot/ash were recovered. Results of the analysis for residual agents and metals per kg ash/soot are given in Table 29.

### **6.12 Residues on Aerosol Filters of the APCU**

All six HEPA filters of the APCU were replaced after the first Phase. No samples were taken at that stage.

During the second Phase, one (of the six) filters was replaced and sampled after 38 detonations. After another 22 detonations, the filter at the same site was replaced and sampled. At the end of test Phase II, the filter at the same place was sampled again,

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<sup>12</sup> By immission is meant: concentration of a contaminant found at a certain point due to emission at another point

together with another filter that was representative for the whole Phase II. Filters were analyzed for residual CWA and total Al, As, Cu and Pb. Results are expressed as g/filter (Table 30). It was seen that, with the exception of As, the quantities on the filter that went through the whole Phase II were roughly (+/-15%) the sum of the three filters that were replaced after the sub-Phases.

### 6.13 Waste Stream

The period of 32 total testing days was split in 21 operating days, during which 90 detonations were realized. Eleven days were used for maintenance, decontamination and clean up. Seventy-eight of the 90 detonations were toxic shells.

Decontamination and clean up resulted in a total waste of 3.2 tons, that can be divided into various parts as given in Table 31. The weight of the protective equipment was not completely included in these figures.

The total weight of ashes/soot recovered from the expansion chamber was 108.1 kg or a mean value of 1.2 kg per detonation.

### 6.14 Estimation of the Mean Quantities Undestroyed

Due to the heterogeneous nature of some samples, such as pea gravel and wipe samples and the unlikelihood of taking representative samples at APCU filters and from ashes/soot in the expansion chamber, it is difficult to calculate exact destruction efficiencies. The following approach was proposed to obtain indicative values.

The aim was to calculate mean quantities of agent not destroyed per shell. Values were calculated from residue analysis in the detonation chamber (pea gravel and wipe), in the expansion chamber (airborne and soot), in the APCU bag house and duct (airborne as well as residue on NORIT filter) and at the exhaust fan.

For DA, the mean quantities destroyed were estimated from the results obtained from evaluating the 15 detonations series on June 15 (Table 19). Residue in pea gravel was calculated from the value of 1235 mg/kg obtained after the 15<sup>th</sup> detonation, divided by 15 (to take into account the cumulative effect of successive detonations) and multiplied by 350 kg, the estimated quantity of pea gravel in the detonation chamber. Residue from the wall was calculated from the value of 115 mg/m<sup>2</sup> (Table 19) and multiplied by the wall surface (Table 2). The airborne residues in expansion chamber and bag house/ductwork were not calculated from values obtained from the same 15 detonations test since no values were measured. Instead, the mean values from the previous series (detonation 18 to 29) were taken and multiplied by the volume of the expansion chamber and bag house plus the ductwork. The contribution of agent adsorbed on dust and soot in the expansion chamber was calculated from a sample taken after detonation 44. This value was multiplied by the mean value of dust/soot produced per detonated shell. The latter was derived from the total weight of soot/dust divided by the total number of detonations (90). In addition, consideration was also taken for the value measured at the outlet of the fan after the charcoal filters. The values obtained during and after the 15 detonations series were multiplied by the respective operation times<sup>13</sup> of the fan and by the airflow of the fan (40 m<sup>3</sup> per hour).

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<sup>13</sup> Obtained from the field diaries.

For the other agents, a similar approach was followed, except for pea gravel values. No cumulative effect was taken into consideration, but only simple mean values were calculated. The results as quantities of undestroyed CWA were summarized in Table 32.

From Table 32, it was concluded that the destruction of CG, PS and HD was quite good. The poor destruction effectiveness of DA and Chlorobenzene was attributed to the inherent chemical stabilities of the molecules and for DA in particular, the specific filling method of the shell (protective effect of the bottle). It should be stressed that these values were the result of analysis of samples taken at several points along the path to the exhaust and that not every value covers neither the same time span, nor the same series of detonations. "Double counting", therefore cannot be excluded.

## **7. CONCLUSIONS AND RECOMMENDATIONS**

### **7.1 Conclusions**

- The Contained Detonation Donovan (detonation chamber, expansion chamber and bag-house with carbon filter system) performed as designed to contain the blast and overpressure from the explosive treatment of CWA shells.
- Air emission levels in ppm-m (CO, Ethylene, Methane, Acetylene, etc.) from the use of oxygen-enriched "sheet" high explosive were observed, as expected, from the exhaust of the carbon filter system.
- Treatment of CWA shells by use of oxygen-enriched "sheet" high explosive was demonstrated; however, residual levels of CWA were observed in the detonation chamber, expansion chamber and the ductwork leading to/from the bag-house.
- A maximum throughput rate of 15 CWA shells was demonstrated during half of a work shift on 15 June 2001.
- Air emissions containing low levels of CWA were observed from the carbon filters during some of the tests. Breakthrough of the carbon filters was attributed to saturation of the filters by extremely high amounts of water vapor as well as the filter setup (varying lengths of ductwork to the individual filters).
- Testing of various additives to the "sheet" explosives in the detonation chamber was not successful in reducing the residual levels of CWA in the detonation or expansion chambers.
- The personnel protective equipment worn by the explosive technicians who performed shell loading and decontamination of the detonation chamber were adequate for the contamination exposure levels.

- Decontamination of the detonation and expansion chambers was difficult and time consuming due to the limited access to the equipment.

## **7.2 Recommendations**

- Design an adequate filtration system capable of handling CWA that can also withstand the high levels of water vapor from the detonation.
- Increase access to the detonation and expansion chambers for ease of decontamination and time reduction.
- Evaluate additional additives to the “sheet” explosives or new explosive types to reduce the residual levels of CWA in the detonation and expansion chambers.
- The US implement a test program to demonstrate/validate the primary treatment system that results from equipment modifications/additions. The primary treatment system shall be optimized to meet specific US applications. Test objectives should include as a minimum: 1) qualitative and quantitative characterization of air emissions and solid waste from the process to 2) ease of decontamination 3) ease of operation and munitions handling 4) personnel protective equipment requirements.

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## Abbreviations/ Acronyms/ Symbols

AC= Ammonium Chloride  
AEL= Airborne Exposure Limit  
Al= Aluminum  
AN= Ammonium Nitrate  
APCU= Air Pollution Control Unit  
As= Arsenic  
ATD= Automatic Thermal Desorption  
BGAD= Blue Grass Army Depot  
CB= Chlorobenzene  
CDC= Contained Detonation Chamber  
CG= Phosgene  
Clark I= diphenylchloroarsine  
Clark II= diphenylcyanoarsine  
Cu= Copper  
CWA= chemical warfare agents  
CWC=Chemical Weapons Convention  
CZE= Capillary Zone Electrophoresis  
D= Exhaust Duct  
DA=Clark  
DAAMS= Depot Area Air Monitoring System  
DBC= Donovan Blasting Chamber  
DBS= Donovan Blast Sheet Explosive  
DC= Detonation Chamber  
DMF=Dimethylformamide  
DNB= Dinitrobenzene  
DOVO= Belgium Bomb Disposal Unit  
EC= Expansion Chamber  
ECBC= Edgewood Chemical Biological Center  
FTIR= Fourier Transform Infrared Spectrometer  
GC/MS=Gas Chromatograph Mass Spectrometer  
GMT= Greenwich Mean Time  
HD= Mustard, Yperite  
HPLC= High Performance Liquid Chromatography  
ICP-AES= Inductively Coupled Plasma Atomic Emission Spectrometry  
LFKGr=Lange Field Kanone Granat  
LOST= HD, named after Germans Lommel and Steinkopf

MC, MINICAMS= Miniature Chemical Air Monitoring System  
MMR= Massachusetts Military Reservation  
NA= Not Analyzed  
ND= Not Detected  
OPFTIR= Open Path Fourier Transform Infrared Spectrometer  
OSHA= Occupational Safety and Health Administration  
PPE= Personal Protective Equipment  
Pb= Lead  
PIC= Picric Acid  
PS= Chloropicrin  
PVC= Polyvinyl Chloride  
QP= Quality Process Samples  
RDX=Trimethylenetrinitramine, hexogen, high brisance explosive  
RMA=Royal Military Academy of Belgium  
RT= Retention Time  
SDM= Service for destruction of munitions  
SIM= Selected Ion Monitoring  
TIC= Total Ion Chromatogram  
TNN= Trinitronaphthalene  
TNT= Trinitrotoluene  
TWA= Time Weighted Average  
USACE= U.S. Army Corps of Engineers  
USEPA= U.S. Environmental Protection Agency  
UXO=unexploded ordinance  
VAC= Volatile Aromatic Compounds  
VCS= Vapor Containment System  
VOC= Volatile Organic Compounds  
WP= White Phosphorus  
WWI= World War I  
XSD= Halogen Specific Detectors



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## **APPENDIX 1.**

### **GENERAL SAMPLING PROCEDURES**

#### **1.1 General Sampling Procedures**

- At the Expansion Chamber (EC) and post-bag house (duct to absolute filter unit) air sampling was achieved by using TENAX (for CWA), charcoal tubes (for VOC, and occasionally for CWA) and ORBO24 (for Phosgene analysis exclusively).
- Systematically, wipes were taken from the inside of the chamber (0.0225 m<sup>2</sup>): pre-extracted, pre-wetted cellulose was used; samples were kept in glass jars.
- Pea gravel was systematically (i.e. following a specific pattern) sampled at the inside of the detonation chamber and collected in a glass jar.
- Particulate matter was sampled by using quartz filters (only during Phase I) and Millipore membranes.
- Soot samples were taken with a spoon. When the soot sticks to the wall or floor, it was scraped off.
- Filter samples: the metal grid was cut with a pair of scissors and two to five folds were cut out of the filter.
- All samples were duly labeled (mentioning the date, sample type, sampling position, sampling time and shell number, if applicable).

#### **1.2 Procedures for set-up/operation /recovery of samples**

##### **1.2.1 GENERAL PRE-TEST AND POST-TEST**

- Complete all data recording forms and confirm accuracy of entries all ready on data sheet.
- Check flow rate of sample at end of each test (before removing sampler) for:
  - a. Millipore 42 mm filter cassette
  - b. TENAX tubes
  - c. Bubbler (impinger)
- Place water bubblers (through exit arm) using 50 ml pipette and remove water by pouring it into clean jar conduct bubbler charging and recovery only in shelter. Record volume of water added.
- In shelter only, place 47 mm quartz filters into filter holder and remove from filter holder and always handle with tweezers. Use separate Petri-dish for each filter. Filters should be weighed before use.
- Sampling ports on expansion chamber: always confirm they are open before starting sampler pumps and closed after stopping sampler pumps.
- Read rotameters when they are vertically straight up and read at center of ball. Rotameters will be used for coarse setting. Final reading of flow will be taken by using bubble flow meter (Gillian).

- Make certain that all pump exhausts (except Gillian Pump) go into exhaust tubing
- Check to confirm all sampling lines are properly connected
- Samples will be recovered after each detonation and properly identified.

### 1.3.1 SPECIFIC PROCEDURES FOR EACH TYPE OF SAMPLER

#### 1.3.1.1 Millipore filter cassette

- Remove caps and install tubing.
- Start pump and collect sample for appropriate time
- Stop pump
- Close sampling port
- Disconnect cassette at inlet; attach flow meter on inlet. Start pump. Note and record flow rate using bubble flow meter.
- Remove filter, check for anomalies, cap ends. Identify properly.
- Install new filter.
- Place used filter in proper container for return to laboratory

#### 1.3.1.2 TENAX sampler

- In shelter, place filter in holder and TENAX tubes in container, record all information needed on data sheet.
- Place filter holder and TENAX tubes in tubing;
- Start pump and collect sample for 50 to 60 minutes. Note and record exact sampling time.
- Stop pump
- Close sampling port
- Disconnect filter at inlet; attach bubble flow meter on inlet; start pump; note and record flow rate
- Remove filter housing and TENAX tube containers, cap off and then return them to shelter for recovery.
- Recover filter and TENAX tube(s) and identify using appropriate label
- Place new filter and TENAX tube(s) in filter holders/containers

NOTE: Protect TENAX tubes from sunlight (heating) at all times, particularly during sample collection

#### 1.3.1.3 Bubblers (Impingers)

- In-shelter: Place 47 mm filter in holder using tweezers and close holder. Use a 50 ml pipette to place distilled water in first two impingers and then connect impinger together. Seal inlet and exit openings
- Move sampler to sampling point and install in sampling line.

- Start pump and sample for 50 to 60 minutes. Note and record exact sampling time.
- Stop pump; close inlet valve and disconnect filter at inlet
- Attach bubble flow meter; start pump.
- Note and record flow
- Move sampler (filter holder, bubbler) to shelter for recovery.
- Remove filter and place in Petri dish. Label dish appropriately.
- Pour contents of impinger into clean jar through impinger exit arm and add 25 ml of distilled water to each impinger. Pour contents into same jar as original impinger contents. Seal and label jar.
- Place clean filter in filter holder and 50 ml into each of first two impingers.

#### **1.3.1.4 TEDLAR bag (Check on purge TEE/on-off valve)**

- Attach TEDLAR bag to pump exhaust
- Start pump, open valve on bag.
- Stop pump when bag is 80-90% full and immediately close valve on bag.
- Remove TEDLAR bag and return to shelter. Protect as much as possible from direct sunlight during sampling.
- Label bag appropriately.

#### **1.3.1.5 Wipe samples**

- Prepare cotton pre-extracted with methanol p.a. in jar
- Wipe 15x15 cm surface using frame
- Put cotton wipe in jar. Close jar
- Label appropriately

#### **1.3.1.6 Charcoal tube and ORBO24™**

- In shelter, place charcoal tube in holder and record all information needed on data sheet.
- Place holder and tubes in tubing;
- Start pump and collect sample for 50 to 60 minutes. Note and record exact sampling time.
- Stop pump
- Close sampling port
- Disconnect filter at inlet; attach bubble flow meter on inlet; start pump; note and record flow rate
- Remove filter housing and charcoal glass tubes, cap off and then return them to shelter for recovery.
- Recover filter and TENAX tube(s) and identify using appropriate label
- Place new filter and TENAX tube(s) in filter holders/containers

**NOTE:** Protect TENAX tubes from sunlight (heating) at all times, particularly during sample collection.

### 1.3.1.7 Pea gravel

Take nine spoon samples in a systematic way as shown in Figure 1.3.1.7.1 below and add to a glass jar.

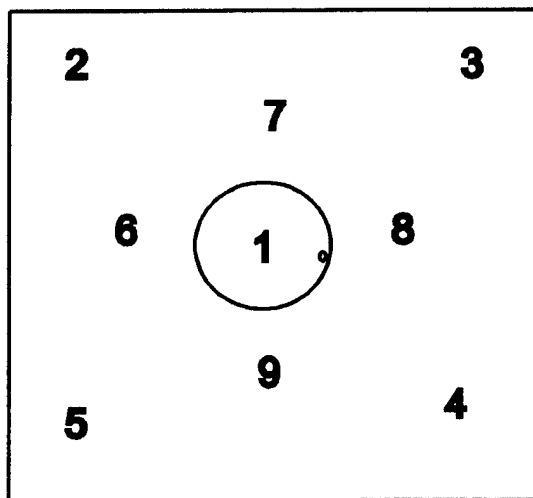


Figure 1.3.1.7.1: Schematic map of the floor of the Detonation Chamber with indication where the spoon samples were taken. All spoon contents were added to a glass jar.

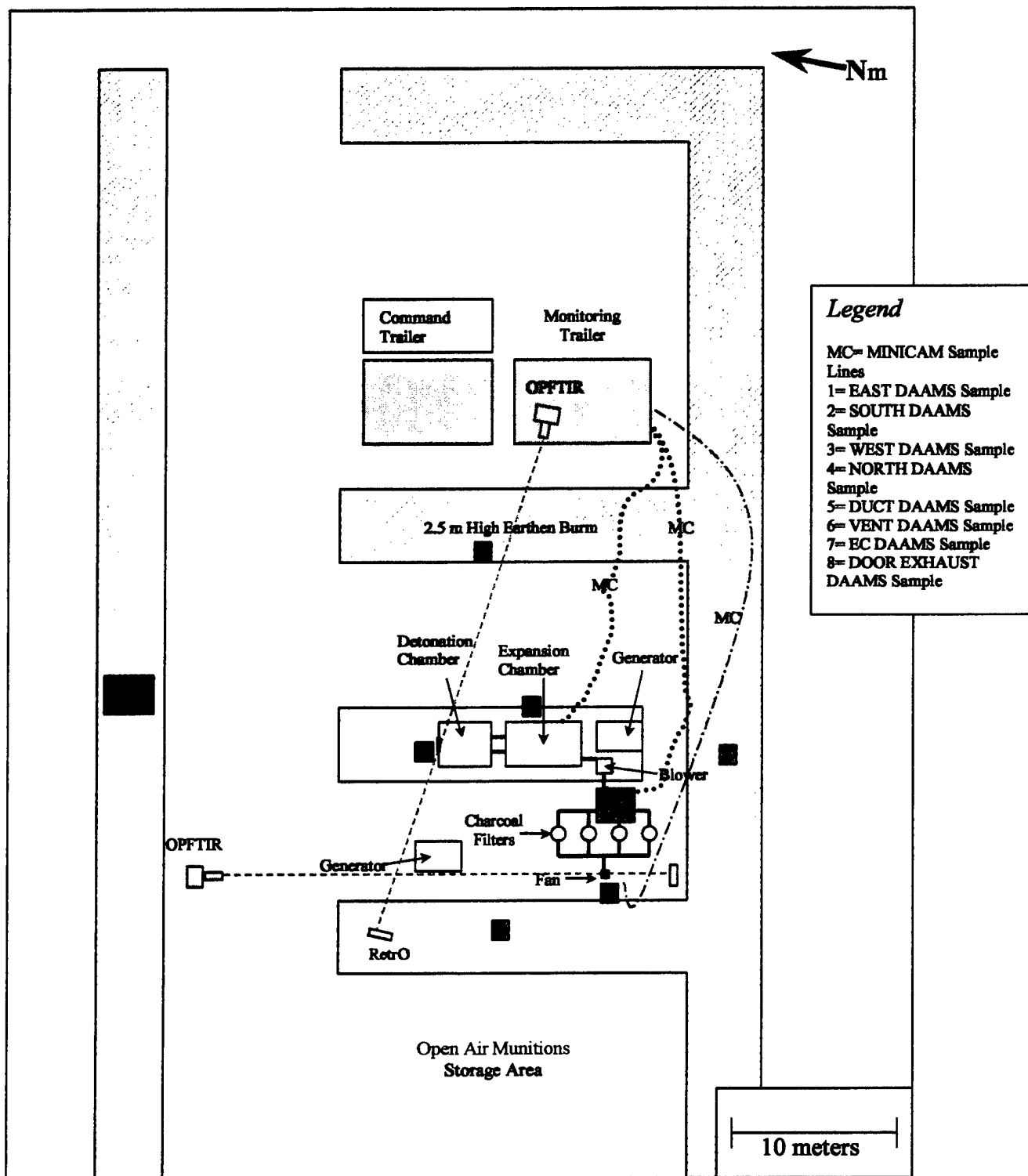
### 1.3.1.8 Depot Area Air Monitoring System (DAAMS) - ECBC

DAAMS, tenax adsorbent filled glass tubes attached to sample pumps were used to collect air samples at various positions around the Detonation chamber during operations. DAAMS were utilized during the Mustard (HD) Phases of the project. Sample positions are included in Table 1.3.1.8.1. Sample positions were changed throughout the project due to limited supply of sample pumps and DAAMS. Positions were sampled on availability of sample apparatus in order of position importance. Sampling time intervals were also varied during the project to correspond to detonation times and to minimize amounts of samples collected on the tube. Quality Process Samples (QP) were used to determine the recovery performance of the DAAMS. A QP is a DAAMS spiked with dilute chemical agent and exposed to sampling environment. QP samples were spiked with 7.2 ng of standard during the Mustard tests.

**Table 1.3.1.8.1 Sampling Positions, Intervals and Flow Rates**

Sample Position (# schematic)	Description	Sample Intervals (min)	Flow Rates (mL)	Volume (L)
Perimeters (1-4) North, South, East, West	10 meters to the north, south, east, and west of chamber	120 240	200 100	24
Expansion Chamber Internal (7)	Attached to sample port near center of expansion chamber	120 60 60 48	200 200 100 100	24 12 6 4.8
Exhaust Duct(5)	Attached to sample port in duct work between particulate and carbon filters	120 240	200 100	24
Expansion Chamber External (7)	Attached to flat bed outside expansion chamber access door	120	200	24
Under Detonation Chamber Door (8)	Attached to flat bed under detonation chamber	120	200	24
Door Exhaust (8)	Attached to sample port on duct work leading from between detonation chamber doors to particulate filters	120 120 240	200 100 100	24 12 24
Exhaust Vent (6)	Attached at vent to environment after carbon filters	240	100	24
Personnel	Attached to chest of chamber operators	120 240	100 100	12 24
Quality Process Sample (QP)	12 meters east of chamber outside of monitoring shed	96	250	24

**Figure 1.3.1.8.1 Schematic of Monitoring around Contained Detonation Chamber**



## APPENDIX 2.

### ANALYTICAL METHODS

#### 2.1 Method A: Assay of Chloride by Capillary Zone Electrophoresis (CZE)(RMA)

##### 2.1.1.1 Sample treatment

Aqueous samples are not treated prior analysis, except for filtering on a 0.45 $\mu$ m membrane. Wipe samples and pea gravel samples are extracted with ultra-high quality water and filtered on 0.45 $\mu$ m membranes.

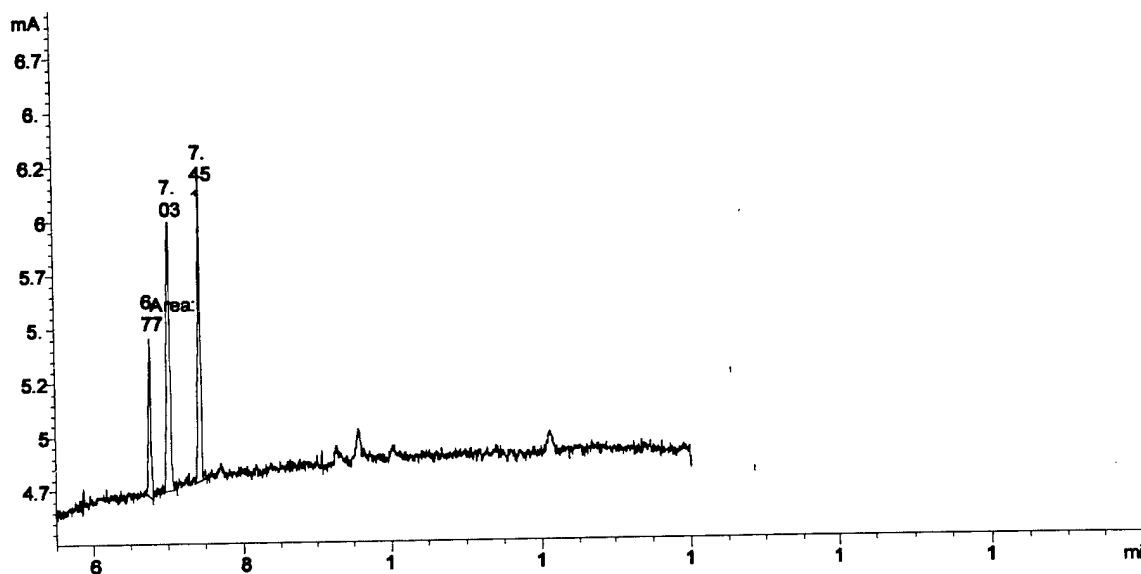


Figure 2.1.1: Electroferogram of a standard mixture. Time scale is in minutes.

##### 2.1.1.2 Analysis

Capillary electrophoresis (CZE mode)

Method used: "Donovan" calibrated method on Br<sup>-</sup> (as external standard) and Cl<sup>-</sup>. Sample is first injected for qualitative analysis followed by 1 standard solution of a mix of 10ppm in the calibrated ions. Successive dilutions to the 0-30ppm ranges of the original sample allow for quantification of the aforementioned ions.

Afterwards spiking of the individual ions reconfirms qualitative presence of the ions detected.

Anion buffer used: -pyromellic acid 2.25mM  
-hexamethonium hydroxide 0.75nM  
-triethanolamine 1.6nM  
-sodium hydroxide till pH7



Capillary used: 50 $\mu$ m fused silica 80.5 cm (72 cm to detector);  
thermostat at 20°C.

Signal DAD: 350nm.

Polarity: reversed polarity (30kV).

Injection: 250mbar.sec

2.1.1.3 Limit of detection=0.1 $\mu$ g/ml

## 2.2 Method B: ICP AES Method (RMA)

### 2.2.1.1 Object

Determine the concentration of aluminum, arsenic, copper, and lead on quartz fiber filters, in wipe samples and in pea gravel.

### 2.2.2.1 Principle of analysis

The samples are digested in a mixture of nitric acid, hydrochloric acid and hydrogen peroxide and an aliquot of the solution is then analyzed by Inductively Coupled Plasma Atomic Emission Spectrometer. The solution is carried into the spectrometer using argon as the carrier gas. In the spectrometer, the elements are brought in excited states by thermal energy (6000-8000 °K). Upon return to the ground state, the energy is transformed in electromagnetic radiation. Each metal emits a characteristic spectrum that allows for qualitative and quantitative determination. Assay is possible via calibration with calibrating solutions of known concentrations of metals. The linear dynamic response of this technique is 4 to 6 orders of magnitude.

### 2.2.2.2 Sample preparation

Samples are dried in an oven at 105 °C. Part of the sample<sup>14</sup> is put in a TEFLON vial and weighed accurately. Six ml HNO<sub>3</sub>, 3 ml HCl and 0.25 ml H<sub>2</sub>O<sub>2</sub> are then added and the vials are closed. Blanks containing only acid/peroxide mixture are added. The vials are sonicated for 15 minutes and treated in microwave oven (MLS-1200 Mega Microwave Digestion System - Milestone Microwave Laboratory Systems). After treatment, the contents of the vials are filtered and quantitatively transferred in 100 ml volumetric flasks. Water is added to the marking.

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<sup>14</sup> Millipore quartz Filters were separated from their plastic casing and transferred to a digestion vial. About one mL of the water samples were filtered over a 0.45  $\mu$ m filter and transferred to a digestion vial; the filter is also transferred to a digestion vial. A quantity of about 0.2 g of dust samples (e.g. from scrape) is transferred to a digestion vial. Pea gravel samples are sieved (1 mm diameter) and about 0.2 g of the sieved material is transferred to a digestion vial. That part of wipes that is visually contaminated is transferred to a digestion vial. About 0.2 g of aerosol filters is transferred to a digestion vial.

### 2.2.2.3 Materials

ICP-emission spectrometer with background correction system  
Pneumatic aerosolisation system with peristaltic pump  
Microwave Digestion System

- Nitric acid, HNO<sub>3</sub> MERCK Suprapur 65 %
- HCl, 30% MERCK, Suprapur
- Hydrogen peroxide, H<sub>2</sub>O<sub>2</sub>, p.a.
- Water , Milli-Q-185 or equivalent
- Gas: argon (quality 5.0)
- Stock solutions (Cd, Cr, Cu, Hg, Ni, Pb, Zn and As): 1000 mg/l

Calibrating solutions are obtained from stock solutions by appropriate dilutions

	solution 1 (mg/l)	solution 2 (mg/l)	solution 3 (mg/l)
Cd	0.05	0.5	5
As, Cr, Cu, Hg, Ni	0.1	1	10
Pb, Zn	0.2	2	20

A blank solution is prepared from pure acid and MilliQ-185-water (same acid concentrations as in calibrating solutions).

### 2.2.2.4 Calculations

By using dedicated software, calibration curves are calculated by plotting the emission intensities versus elemental concentrations. From the measured intensity in a sample, the corresponding concentration can be calculated.

### 2.2.2.5 Detection limits

Detection limits are given in table 1. These limits are valid for mass sample quantities of 1 g, digested as described above and diluted to a final volume of 100 ml. Detection limits for bubblers are calculated from the respective detection limits in ppm and transformed to mg/m<sup>3</sup> (by using a typical water volume of 70 ml and a typical samples air volume of 70 liters)

Table 2.2.2.1: Detection limits for the elements analyzed, in mg per kg sample for mass samples or in mg/m<sup>3</sup> for airborne samples

Element	Detection limit (mg/kg)	Detection limit (mg/m <sup>3</sup> )
Arsenic	10	0,043
copper	2	0,214
Lead	14	0,043
Aluminum	4	0,321

### 2.3 Method C: Assay of chemical warfare agents in air by sampling on TENAX and quantitation by ATD-GCMS (RMA)

#### 2.3.1 Object

Air containing chemical warfare agents is collected at 1L/min using TENAX-TA adsorbent tubes. TENAX-TA is a porous polymer that is based on 2,6-diphenyl-p-phenylene oxide. It is used as a trapping adsorbent for volatile organic and semi-volatile organic compounds. The organic compounds adsorbed on the TENAX are then thermally desorbed onto a cold-trap (ATD) and the trapped material is injected by flash heating into a capillary gas chromatography (GC) equipped with Mass Selective Detection (MSD). The target compounds are Phosgene, Chloropicrin, Chlorobenzene, Mustard (Yperite), Diphenylchloroarsine (CLARK I) and Diphosgene.

#### 2.3.2 Reagents and equipment

☐ Solvent(s): Standards are prepared in appropriate solvents: dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) for Chloropicrin, Chlorobenzene, Yperite and Diphosgene; acetone for CLARK

☐ Analyte standards :

For every compound one standard solution (1 ml/100 ml) and several dilutions are prepared.

Phosgene : commercial solution (20 % in toluene)

1 ml / 100 ml: conc. =  $0.2 \times 10000 \times 0.935$  (=density<sub>Phos</sub>) = 1870 mg/l

Chloropicrin : >98 % pure (commercially obtained from ...)

1 ml / 100 ml : conc. =  $10000 \times 1.65$  (=density<sub>Clpicrin</sub>) = 16500 mg/l

Chlorobenzene : anhydrous, 99.8 %

1 ml / 100 ml : conc. =  $10000 \times 1.107$  (=density<sub>Clbenzene</sub>) = 11070 mg/l

Mustard gas (HD) : > 99% pure

1 ml / 100 ml : conc. =  $10000 \times 1.27$  (=density<sub>HD</sub>) = 12700 mg/l

Diphenylchloroarsine (CLARK D):

ca 0,1 g is weighed and dissolved in 100 ml acetone  
conc. =  $0.1 \times 10000 \times 0.92$  (= purity after distillation)

Diphosgene: 99 % pure (commercially obtained from ...). 1 ml / 100 ml: conc. =  
 $1 \times 10000 \times 1.6525$  (=density<sub>Diphos</sub>) = 16525 mg/l

- ☐ Carrier gas: helium, chromatographic quality
- ☐ Special precautions:  
All compounds mentioned are toxic and irritating.  
Mustard (Yperite) is a human carcinogen.  
Dichloromethane and toluene are suspected carcinogens.

Sampling equipment:

TENAX sorbent tubes were obtained from PERKIN-ELMER. These tubes can be reconditioned and are re-usable. Reconditioning of TENAX tubes is done by purging with a helium flow of 30 ml/min at a temperature of 320 °C for at least 30 min.

Analytical equipment:

Automatic Thermal Desorption System (ATD), PERKIN ELMER  
Gas chromatograph with column (DB-5MS, 30m x 0.32 mm ID x 0,5 µm df)  
coupled to a Mass Selective Detector and Chemstation Data System (HEWLETT  
PACKARD 5890 Series II)

### 2.3.3 Methodology

Automatic Thermal Desorption/Cold Trapping, followed by flash-heating injection into a gas chromatograph and detection by electron impact mass spectrometry, with selected ion monitoring:

- ☐ Thermal Desorption – Cold trapping :  
Desorption at 300 °C during 10 min, desorb flow = 3 – 20 ml/min (He-gas)  
Cold trapping at – 30 °C  
Flash heating : 40 °C/ s
- ☐ Chromatographic conditions:  
Injector temperature: 180 °C ; Detector temperature : 260 °C  
Oven program: 35 °C during 5 min ; Rate = 10 °C/ min till 200 °C  
Column: DB-5MS (95% methyl, 5% phenylpolysiloxane)  
30m x 0,32mm ID x 0,5 µm df  
Carrier gas: Helium 50 kPa ; Column flow = 2,78 ml/min  
Split or splitless, depending on the estimated amount on the tube.

□ **Selected Ion Monitoring (SIM)**

Chloropicrin:	ions: 82, 84, 117, 119 and 12 RT interval = [4.00 – 6.50]
Chlorobenzene:	ions: 77, 112 and 114 RT INTERVAL = [6.50 – 8.00]
Yperite (HD)	ions: 63, 109, 111 and 158 RT INTERVAL = [11.0 – 15.0]
Diphenylchloroarsine:	ions: 154, 51, 77, 187 and 227 RT INTERVAL = [15.0 – 18.00]
Diphenylcyanoarsine:	ions: 154, 51, 77, 101 and 125 RT INTERVAL = [15.0 – 18.0]
Phosgene:	ions: 63, 65, 35, 98 and 100 RT INTERVAL = [1.00 – 4.00]
Diphosgene:	ions: 59, 117, 119, 63, 65 and 113 RT INTERVAL = [8.00 – 11.0]

The selected ions of each compound are scanned within the given retention time interval.

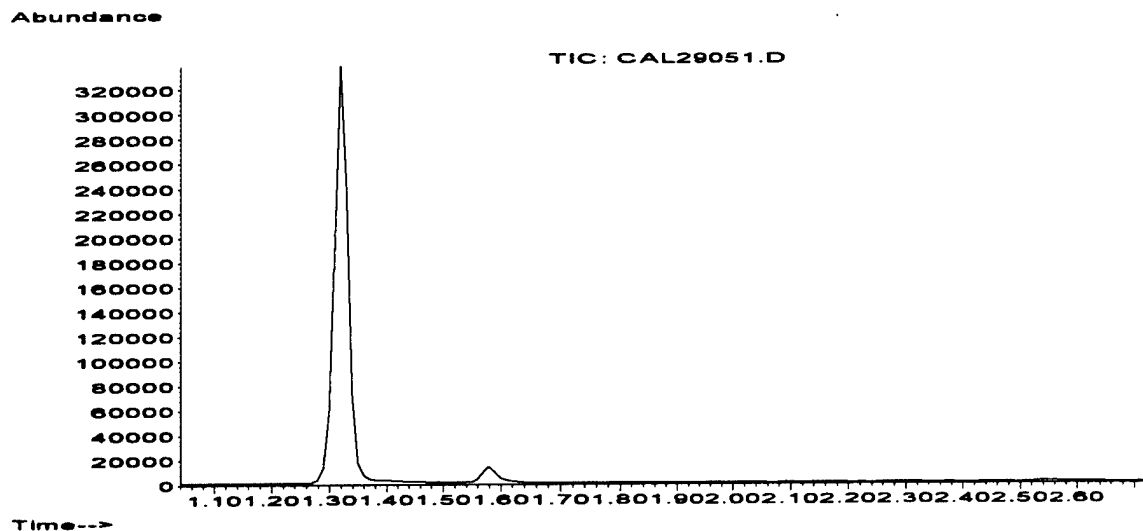


Figure 2.3.3.1: Typical chromatogram (TIC) for Phosgene calibration standard. Time scale is in minutes.

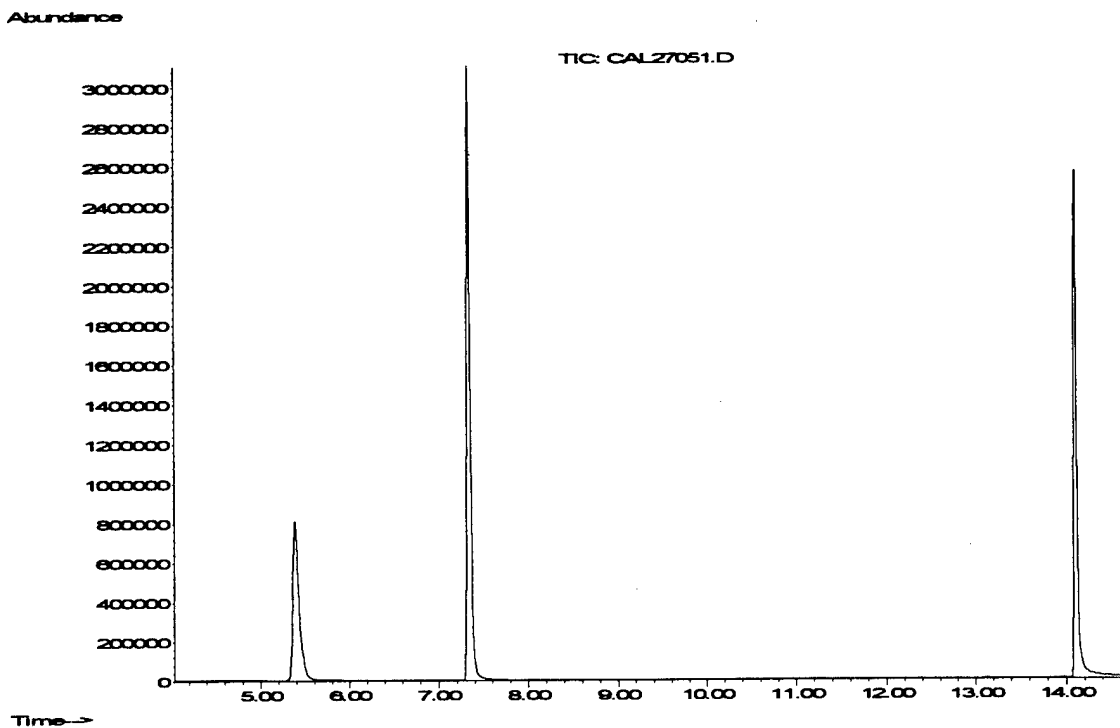


Figure 2.3.3.2: Typical chromatogram (TIC) for Diphosgene calibration standard. Time scale is in minutes.

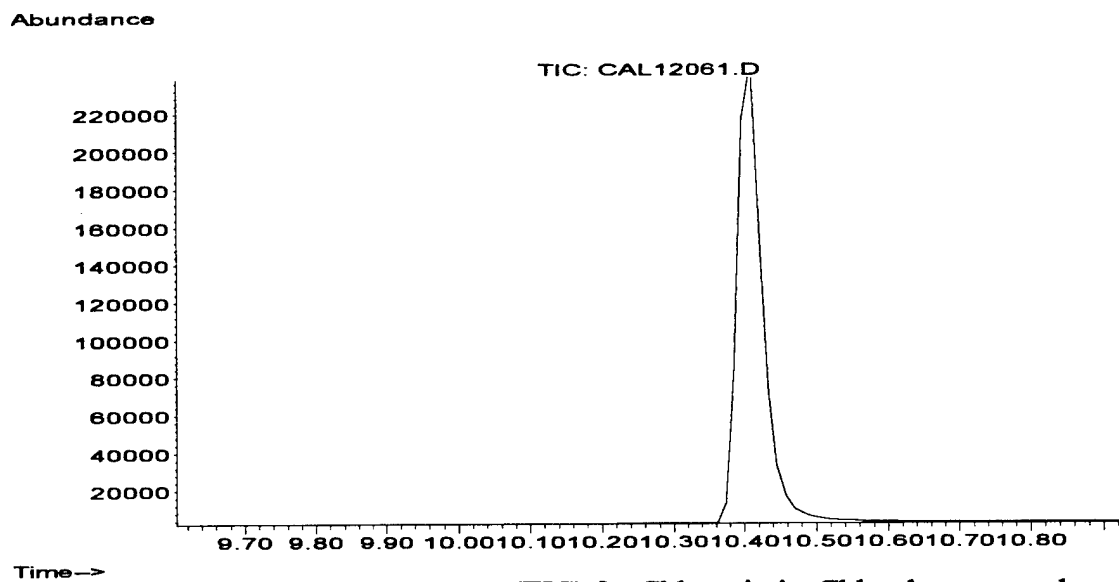


Figure 2.3.3.3: Typical chromatogram (TIC) for Chloropicrin, Chlorobenzene and Mustard calibration standards. Time scale is in minutes.

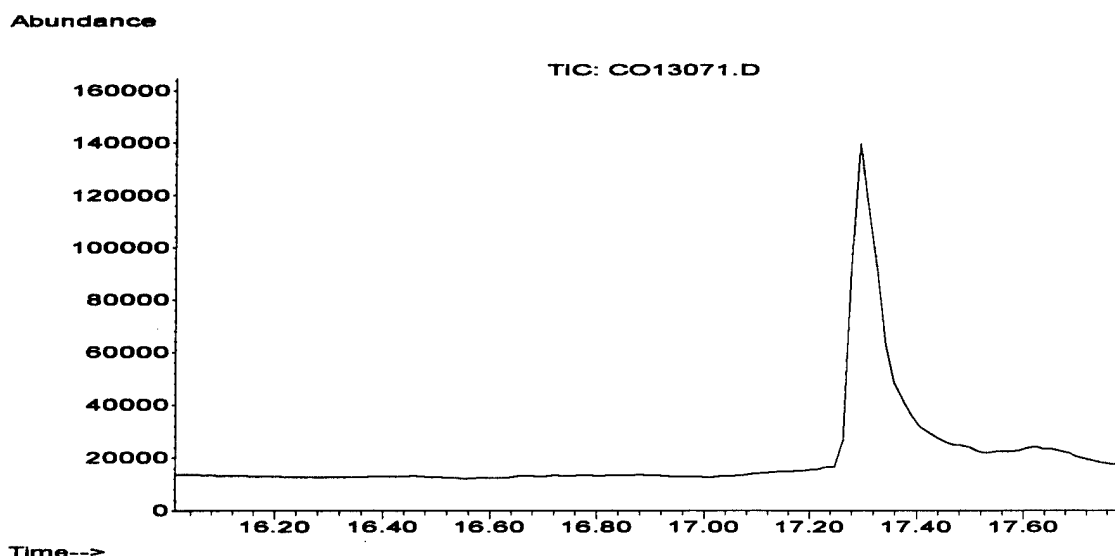


Figure 2.3.3.4: Typical chromatogram (TIC) for Diphenylchloroarsine CLARK I calibration standard. Time scale is in minutes.

#### 2.3.4 Calibration

The quantity of compound on the TENAX-tube (ng) was determined by a calibration plot (amount [ng] in ordinate and response [SIM peak height]). For every compound, a control standard with known concentration should be analyzed every day to verify if the calibration plot shows no deviation.

The calibration plot is made up within the range of 1 till 100 ng. Various standard solutions, containing the analytes are prepared and various volumes (1 up to 5  $\mu$ l) of those dilutions are injected on pre-conditioned TENAX-tubes and analyzed as described previously. The flow to the injector can be "split" to avoid saturation of the detector.

#### 2.3.5 Detection limits

Absolute detection limits (in ng) at the detector are determined as three times the noise level just before elution of the chromatographic peak and by converting this noise level to a quantity of agent via the calibration line.

#### 2.3.6 Method detection limits

Method 1, for on-column injection of the total contents of the sorbent tube. This method is, as a rule used for airborne samples. The method detection limits are expressed in  $\text{mg}/\text{m}^3$  and are calculated from the absolute detection limits by taking into account a representative value for the sampled volume (1L) and converting to the units used.

Method 2, for pea gravel extract. A typical sample weight of 0.5 kg is extracted in appropriate solvent and the extract is reduced to 10 milliliters; one microliter is injected –via a sorbent tube- on column.

Method 3, for wipe samples. A sample representing a surface of 0.0225 m<sup>2</sup> is extracted in appropriate solvent and the extract is reduced to 10 milliliters; one microliter is injected –via a sorbent tube- on column.

Table 2.3.6.1: Detection limits for various agents

Compound	Absolute Detection Limit (ng)	Method 1 Detection Limit (µg/m <sup>3</sup> )	Method 2 Detection Limit (µg/kg)	Method 3 Detection Limit (µg/m <sup>2</sup> )
Yperite	0,0093	0,01	0,2	4,1
Chlorobenzene	0,0015	0,002	0,03	0,7
Phosgene	0,017	0,02	0,3	7,6
Diphosgene	0,011	0,01	0,22	4,9
Chloropicrin	0,092	0,09	1,8	41
CLARK I	13,2	13	264	5867

### 2.3.7 Results

The results are calculated as mass concentration (mg/m<sup>3</sup> or mg/kg) or surface concentration.

### 2.4 Method D: Gas Chromatograph/Mass Spectrometer (GC/MS) w/ Dynatherm Thermal Desorption Unit for DAAMS (ECBC)

DAAMS tubes were thermally desorbed using a Dynatherm ACEM 900 and analyzed on a 5890 series Gas Chromatograph/ 5972 Mass Spectrometer. The GC was equipped with an Agilent 1701 (14% cyanopropylphenol/ 86% dimethyl polysiloxane) capillary column. The GC/MS was used during the Mustard detonations. The instrument was calibrated using a 1.8 ng/µL Mustard (HD) in hexane standard. Due to the limited standard concentrations many results were calculated assuming HD was linear. Confirmation samples were analyzed in a Selected Ion mode (SIM) and contaminated samples were analyzed in a full scan mode to examine other products such as Chlorobenzene, 1,4-Oxathiane, and 1,4-Dithiane.



**Table 2.4.1 Dynatherm Parameters**

Temperature Settings	(C°)	Time Settings	(min)
Valve	190	Dry	1
Transfer line	240	Heat	3
Tube Heat	290	Trap	2
Trap Heat	300		
Tube Idle	90		
Trap Idle	70		

**Table 2.4.2 SIM Method Parameters**

Oven	MSD
Initial Temperature: 90 °C	Solvent Delay: 4.3 min
Initial Time: 1.00 min	Ions: 109,111,158,160
Ramp 40°C/min	MS Quad: 150°C
Run time: 6.25 min	MS Source: 230°C

**Calibration**

Retention time: 4.8 min  
 Level 1=1.8 ng  
 Level 2=3.6 ng  
 Level 3=5.4 ng  
 Level 4= 9.0 ng  
 Level5=12.6 ng

**Table 2.4.3 SCAN Method Parameters**

Oven	MSD
Initial Temperature: 40 °C	
Initial Time: 1.00 min	Ions: 40-300
Ramp 10°C/min	MS Quad: 150°C
Run time: 24.0 min	MS Source: 230°C

**2.5 Method E. Miniature Chemical Air Monitoring System (MINICAMS) (ECBC)**

MINICAMS were utilized during Mustard (HD) and Phosgene (CG)/Chloropicrin (PS) detonations. MINICAMS, miniature gas chromatographs were equipped with halogen specific detectors (XSD) attached to 100 foot heated

sample lines. MINICAMS were used to monitor the expansion chamber, exhaust duct, exhaust vent, and the monitoring shed during the project. MINICAMS were calibrated daily and challenged every four hours with standard amounts equaled to one TWA (time weighted average) or one AEL (airborne exposure limit). Quality Process samples (QP) were also generated daily to confirm effectiveness of heated sample lines. MINICAMS were capable of providing results every ten minutes for HD and every four minutes for PS/CG.

Table 2.5.1 MINICAMS Parameters for HD

Temperatures (C°)	Ambient	40
	Inlet	100
	XSD Block	150
	Column, low	50
	Column, High	200
	Column, rate	230
	PCT heater, low	50
	PCT heater, high	250
Pressures (psi)	Air	15
	Nitrogen	40
	Hydrogen	15
Sample	Flow rate (ccm)	450
	Volume (L)	2.89
Time (sec)	Purge	0-240
	Desorb	20-70
	Column purge	225
	Sample	240-600
	Column	70-225
	Inject	240-250

Table 2.5.2 MINICAMS Parameters for PS/CG

Temperatures (C°)	Ambient	40
	Inlet	40
	XSD Block	150
	Column, low	40
	Column, high	180
	Column rate	100
	Loop heater	40
Pressures (psi)	Air	15
	Nitrogen	20
	Hydrogen	15
Sample	Flow rate (ccm)	100
	Volume (L)	0.10
Times (sec)	Purge	0-170
	Desorb	0
	Column purge	120
	Sample	170-225
	Column	30-120
	Inject	170-180

## 2.6 Method F: Analysis of Phosgene in Air by adsorption and *in situ* derivatization (RMA)

### 2.6.1. Object

Phosgene in air is collected at 1L/min by adsorption onto ORBO™24 solid sorbent tubes coated with 2-(hydroxymethyl) piperidine. The resulting derivative of Phosgene and coating (figure 1) is extracted in toluene/DMF and analyzed by gas chromatography with Mass Selective Detection. The method is adapted from OSHA Test Method 61.

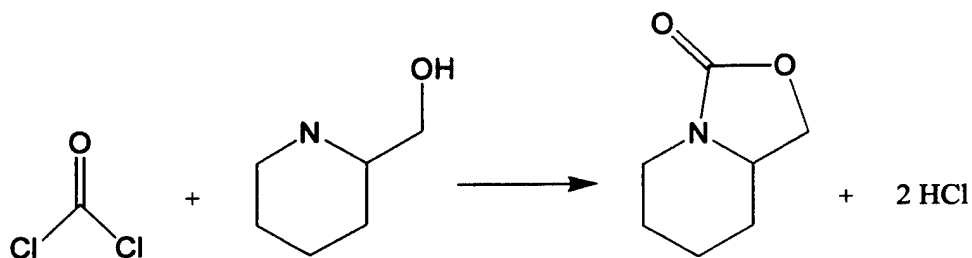


Figure 2.6.1.1: Sorbent reaction on the ORBO™24 of 2-(hydroxymethyl) piperidine and Phosgene, resulting in formation of a derivative hexahydro-oxazolo[3,4-a]pyridin-3-one.

## 2.6.2 Reagents and equipment

### Reagents

*Solvent* : Toluene and DMF chromatographic quality

*Analyte standard*: Commercial mixture of 100 ppmv of Phosgene in N<sub>2</sub>

*Carrier gas*: Helium chromatographic quality

*Special precautions*: Phosgene is extremely toxic. Toluene is a suspect carcinogen

### Equipment

### Sampling

Glass tube containing two sections of XAD-2 adsorbent coated with 2-(hydroxymethyl) piperidine (150 mg/ 75 mg) separated by urethane foam plug. (ORBO™24 small tubes)

Personal sampling pump – GILIAN.

### Analysis

Gas chromatograph (AGILENT 6890) equipped with mass selective detector (AGILENT 5973) and data system; column (JW DB5, 30 M, 0.32 mm, 0.5 um)

## 2.6.3 Analytical methodology

Front and back sorbent sections of the sampler are placed in separate vials; glass wool and foam plugs are discarded and 1 ml solvent (20ul DMF/100 ml Toluene) is added. Vials are crimp-capped immediately. Vials are allowed to stand for at least 60 min with occasional agitation. Samples are filtered with 20µm disposable filters (CHROMAFIL) before injecting 1 µL onto the capillary column.

### Chromatographic conditions

Injection volume: 1 µl

Injector temperature: 180°C; detector temperature : 280°C

Carrier gas: Helium (25 kPa/ flow: 18.1 ml/min/split ratio: 10.4:1, split flow: 13.4 ml/min)

Temperature program: 120°C for 1 min, then 10°C/min to 160°C, then 60°C/min to 225°C.

Substance	Retention time (min)	Ion monitored
CG derivative	6.45	139

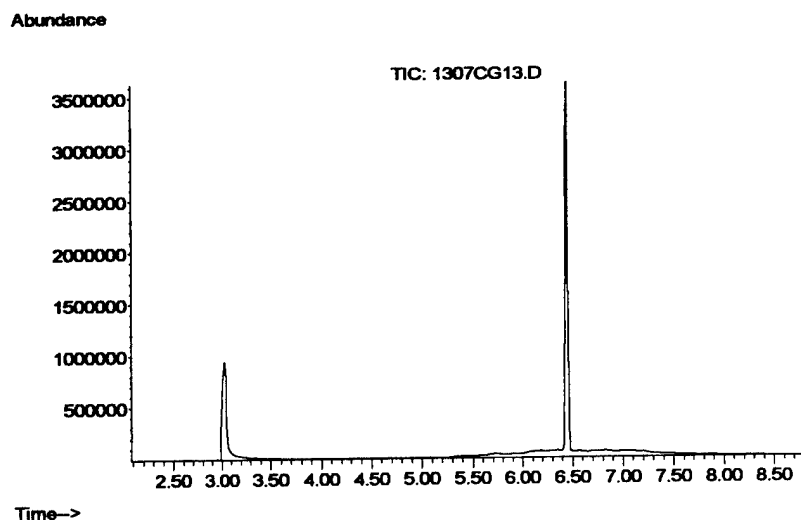


Figure 2.6.3.1: Selected ion chromatogram ( $m/z = 139$ ) of hexahydro-oxazolo [3,4-a]pyridin-3-one. Conditions are as described in the text.

#### 2.6.4 Calibration

Known volumes of a 100 ppmv Phosgene /air mixture are drawn over ORBO™24 tubes through a GILIAN personal sampling pump. One mole= 24L at 293 K  
A calibration graph (peak area of Phosgene derivative vs.  $\mu\text{g}$  Phosgene adsorbed on the tube) is made.

The resulting linear regression is statistically treated: all points outside a 95% confidence interval are rejected.

Because almost all values fall below the lowest calibration point, it was chosen to accept a calibration line of the type  $y = ax$ , i.e. going through the origin. The details of the calibration line are given in Appendix 1.

#### 2.6.5 Detection limits

The absolute detection limit (ng) at the detector is determined as three times the noise level just before elution of the chromatographic peak and by converting, via the calibration line, this value to a quantity of agent.

The method detection limit ( $\mu\text{g}/\text{m}^3$ ) is calculated from the absolute detection limits by taking into account a representative value for the sampled volume (4 liters) and converting to the units used.

Compound	Absolute Detection Limit (ng)	Method Detection Limit ( $\mu\text{g}/\text{m}^3$ )
Phosgene derivative	0.6	0.15

### **2.6.6 Calculations**

Determine the quantity of the analyte found in the sample front ( $w_f$ ) and back ( $w_b$ ) sorbent section by comparing the peak areas for the samples to the calibration curve.

### **2.7 Method G: Open Path Fourier Transformer Infrared Spectrometer (OPFTIR) (ECBC)**

The OPFTIR was used to provide real time monitoring (every 20 seconds). The instrument was composed of a Fourier Transform Infrared (FTIR) Spectrometer, a retro reflector, and a field computer. The OPFTIR was important because it generates an infrared beam of energy in the space between the spectrometer and the retro reflector, which can transform into an electrical response. It was utilized for monitoring for releases above the door of the detonation chamber and at the exhaust vent. The OPFTIR was setup to monitor during the detonations of all munitions tested. The instrument was capable of detecting over 200 different compounds.

### **2.8 Method I: Analysis of volatile aromatic hydrocarbons by GC-MS (RMA)**

#### **2.8.1 Materials and Method**

#### **2.8.2 Reagents**

The analytes used for the calibrations are GC standard quality  $\geq 99.5\%$  (FLUKA). Helium utilized as carrier gas for the GC is He 5.0 (chromatographic quality). The carbon disulfide is HPLC quality (ACROS).

#### **2.8.3 Equipment**

##### **2.8.3.1 Sample treatment**

Sampling is made with glass tube containing two sections of activated coconut shell charcoal (100mg/50mg) separated by urethane foam plug. (ORBO32 small tubes/ Supelco 20267-U) and personal sample pumps (GILLIAN).

The front and back sorbent sections are placed in separate vials and 1 ml of  $\text{CS}_2$  is added to each vial; the vials are immediately closed with caps. Vials are allowed to stand for at least 30 minutes, with occasional agitation. The vials are opened, filtered on 20  $\mu\text{m}$  filters.

### 2.8.3.2 Analytical equipment.

Gas Chromatograph (AGILENT 6890A(G1530A)) equipped with a mass selective detector (AGILENT G2578A), an integrator and a capillary column (JW DB5, 30 M, 0.32 mm, 0.5  $\mu$ m).

- Injection volume : 1  $\mu$ l
- Injector temperature : 220°C; detector temperature : 280°C
- Vector gas : Helium
- Flow rate: 9.1 ml/min
- Temperature program: 40°C for 4 min, then 6°C/min to 150°C, then 50°C/min to 250°C.

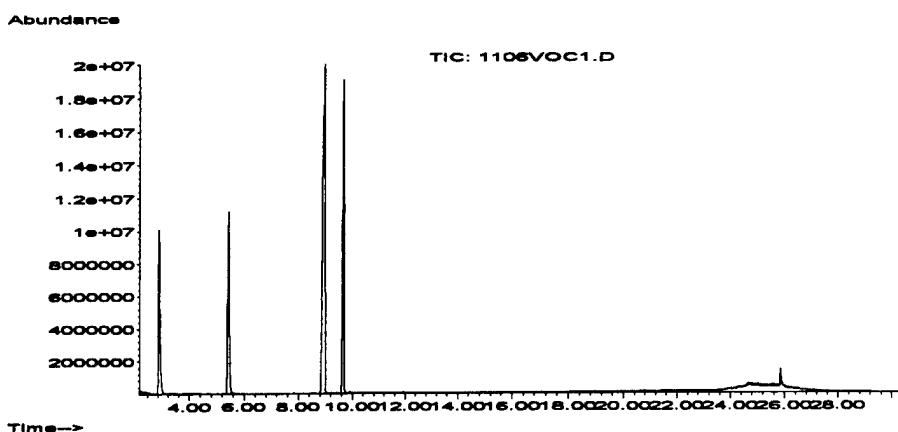


Figure 2.8.3.1: Selected Ion Monitoring Chromatogram of the analysis of a mixture of volatile aromatic hydrocarbons. Identification of the peaks can be done from the retention timetable below.

Substance	Retention time (min)	Ion monitored
Benzene	2.84	77
Toluene	5.45	91
m-p-xylene	8.94	105, 9
o-xylene	9.66	105, 9

### 2.8.4 Calibration

The calibrations are made in the same conditions that those utilized for the unknown samples. The calibration is made with quantity of Phosgene adsorb on the tube.

A mother solution is obtain by adding known amount (10  $\mu$ l) of the analyte to CS<sub>2</sub> in 10 ml volumetric flasks and dilute to the mark. This mother solution is diluted to obtain the calibration mixtures. The mass concentrations are calculated from density data.

#### **2.8.5 Calculations**

The quantity of VOC (benzene, toluene, xylenes) adsorb on each sample is calculated with the calibration curve. The calculations are made on the peak area. The peak area is related to the sample's concentration with the calibration curve equation.



**Blank**

### **APPENDIX 3.**

#### **EVALUATION OF ACTIVATED CARBON FILTER BREAKTHROUGH**

Chemical agent concentrations exiting the exhaust vent of the carbon/HEPA filter system were measured. Since evaluation of breakthrough was not an initial aim, not all values needed for such an evaluation were obtained. Additionally, flow rates through the individual filters were not measured. Only concentrations before and after the filters were available, which made it impossible to obtain a mass balance over the filters. The available results can only be interpreted in a qualitative way.

During each explosion the filters were exposed to extremely high concentrations of water vapor. About 100 bags of water were used during the complete testing campaign. This amounts to approximately 50 kg of water, superimposed on the normal relative humidity of the ambient air at the test site.

It is possible that the filtration flow was not evenly distributed over the four filters. In that case, the air flow over some of the filters was too high, resulting in a dead volume, larger than the actual bed depth, leading to an immediate breakthrough. It was also likely for these two filters to reach their saturation level much sooner.

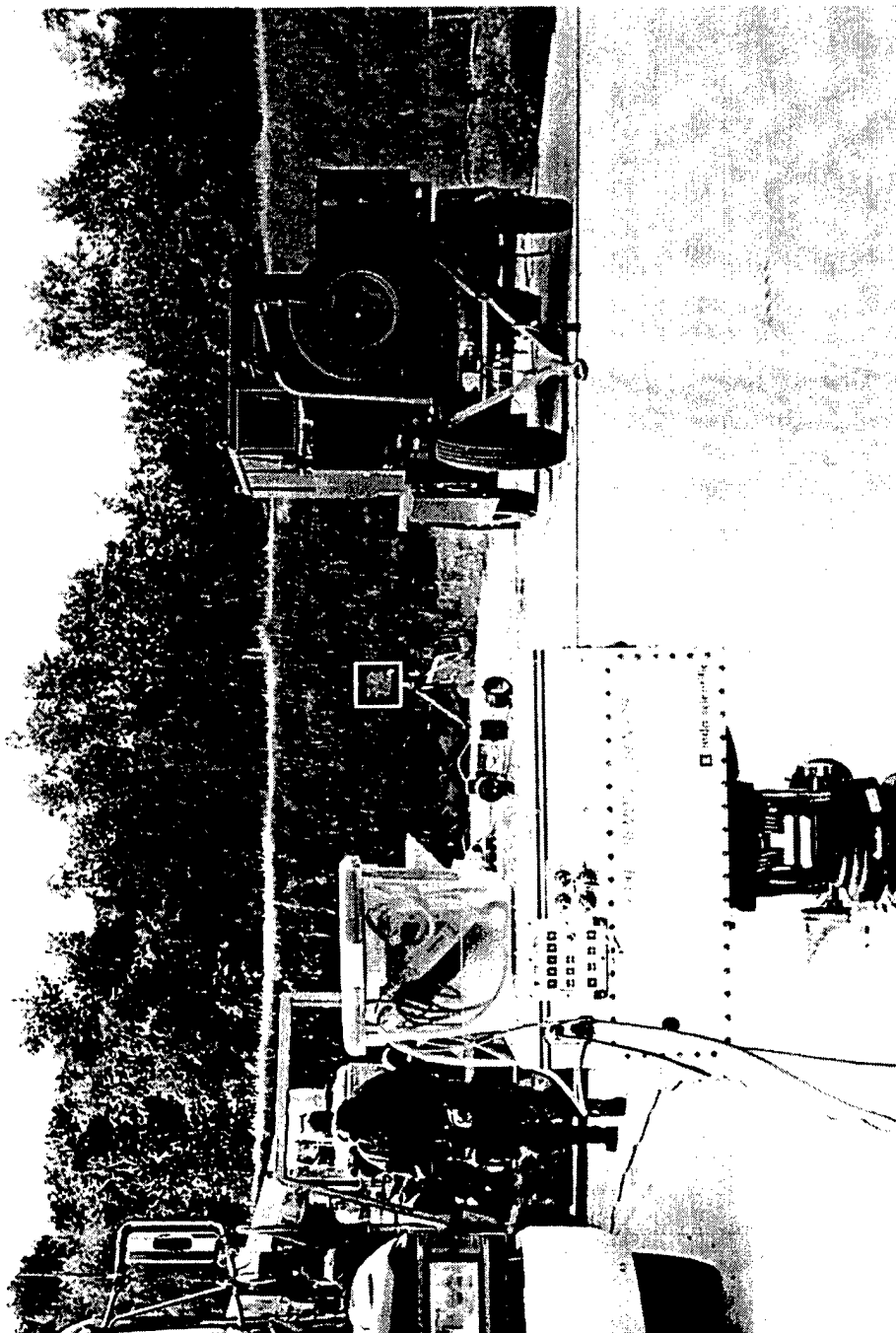
Additionally, the carbon will have suffered from a diminished capacity for the more volatile compounds due to the presence of the adsorbed water. This also means that any given substance with a lower vapor pressure was capable of competing successfully with these compounds. As the filters were completely saturated (essentially with water), every additional adsorption resulted in a release of the most volatile component(s) previously adsorbed on the filter.

The available data is incomplete but is generally consistent with the effects of a saturation of the filters by extremely high amounts of water vapor. Such amounts are indeed released by the water bags used. This adsorbed water will slow down the adsorption process to such extent that the filters present an almost immediate breakthrough of the toxic compounds, even at low airborne concentrations of these compounds and for products with a very low volatility, e.g. Mustard and Diphenylchloroarsine.

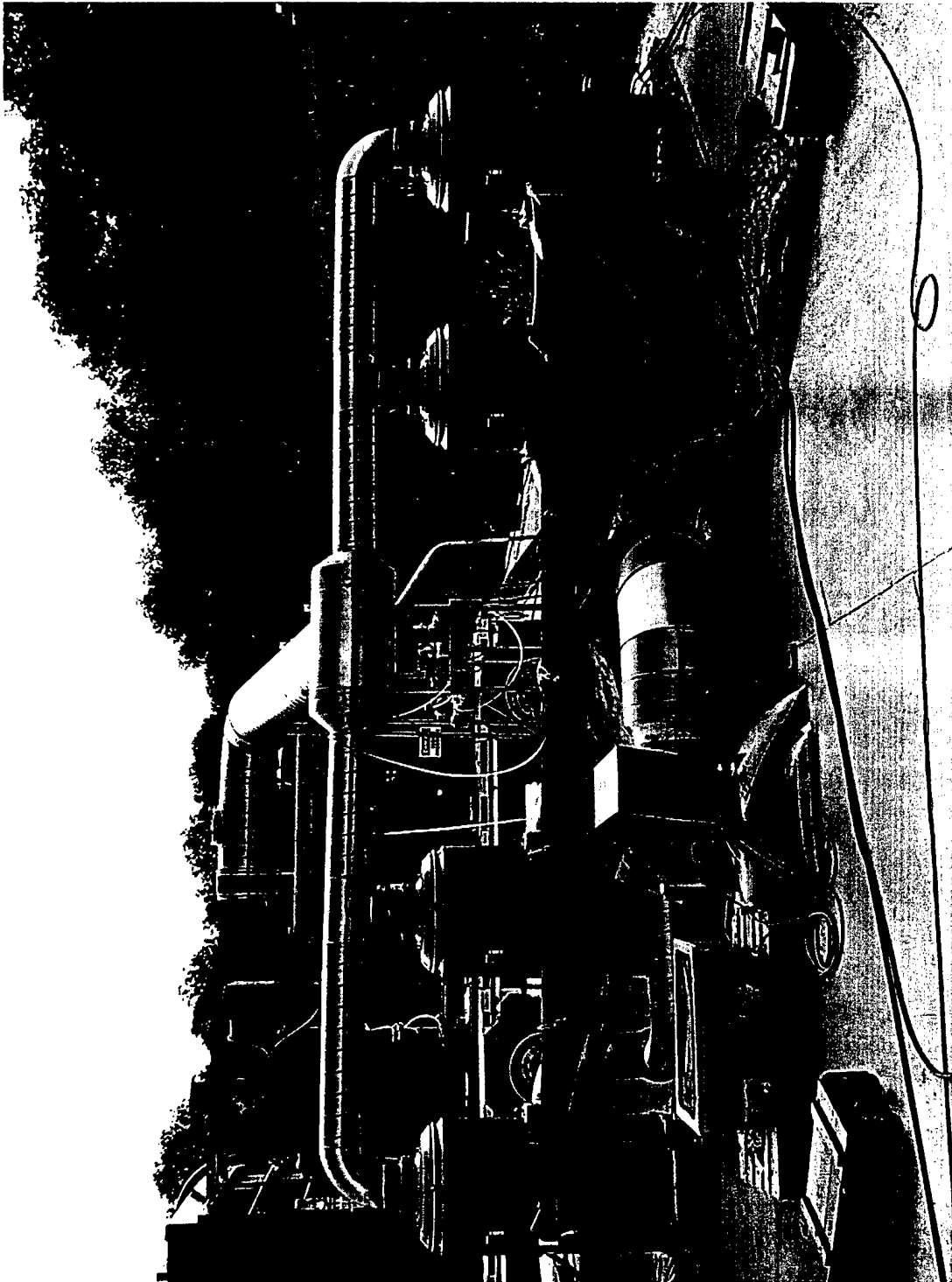
**Blank**

**APPENDIX 4.**  
**PHOTOGRAPHS OF SITE**

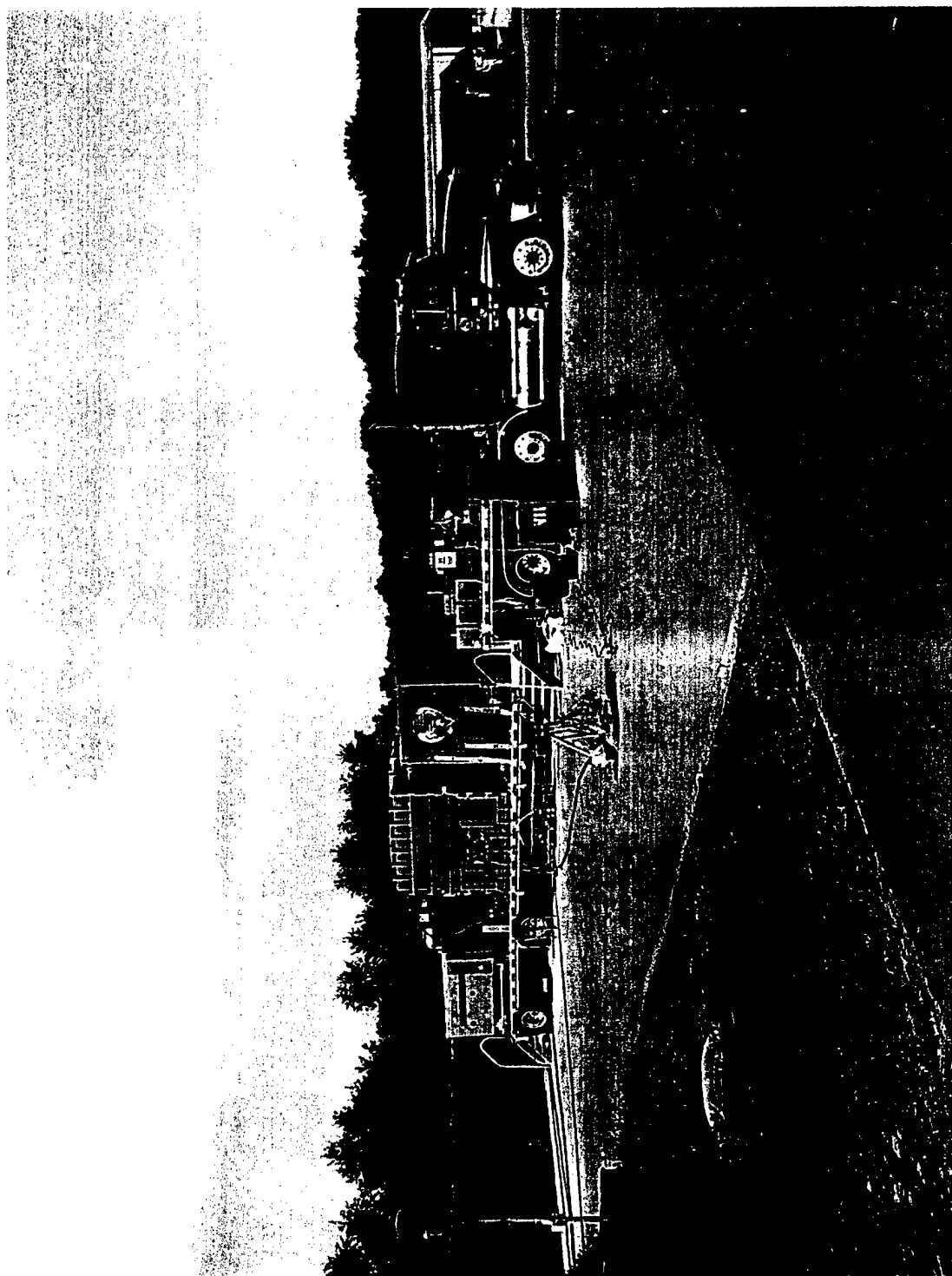
#### 4.1. OPFTIR Set-Up at Exhaust Vent



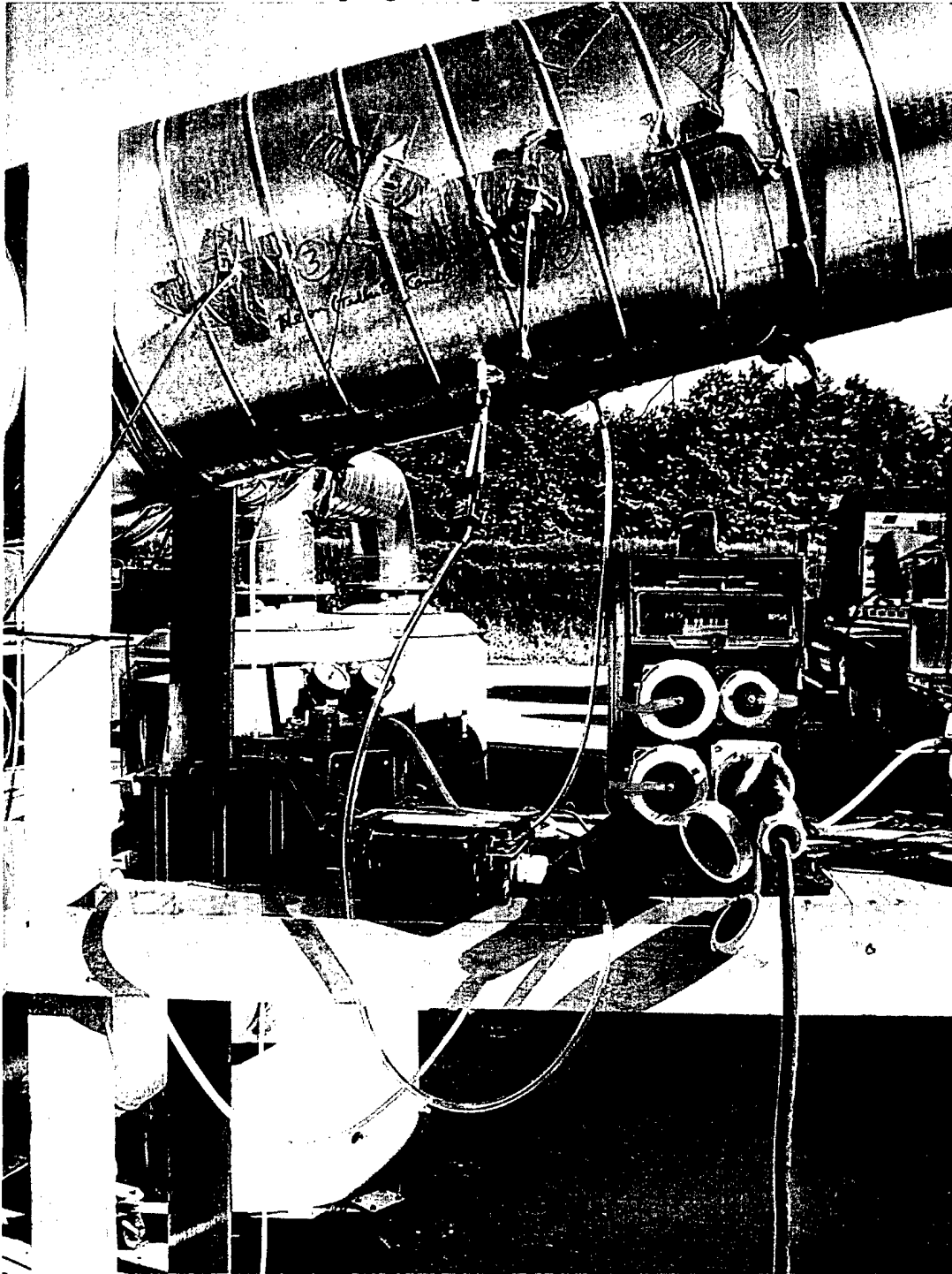
#### 4.2. Exhaust Vent and Carbon Filters



#### 4.3 Southwestern View of Donovan Chamber

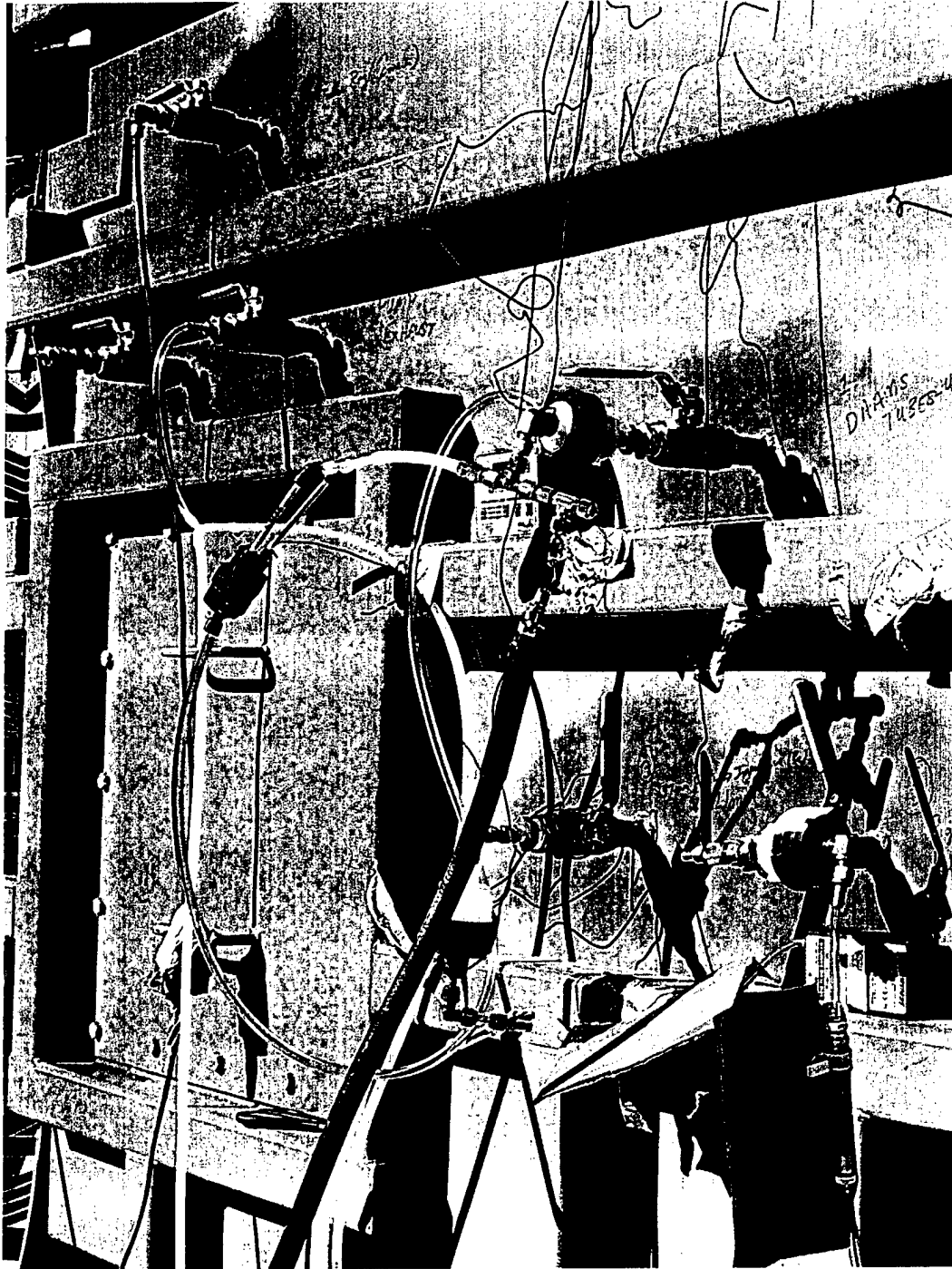


#### 4.4 Sampling Set-Up at the Exhaust Duct

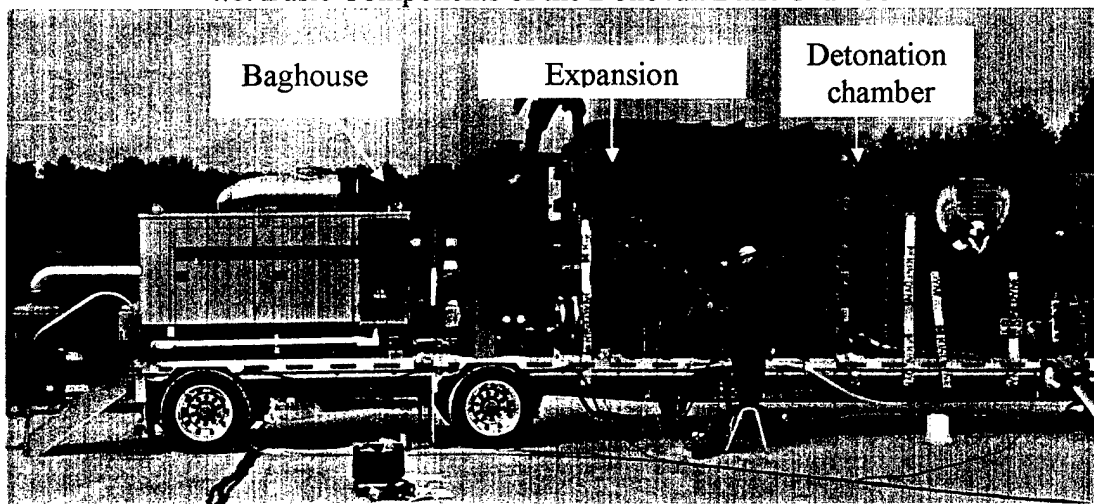




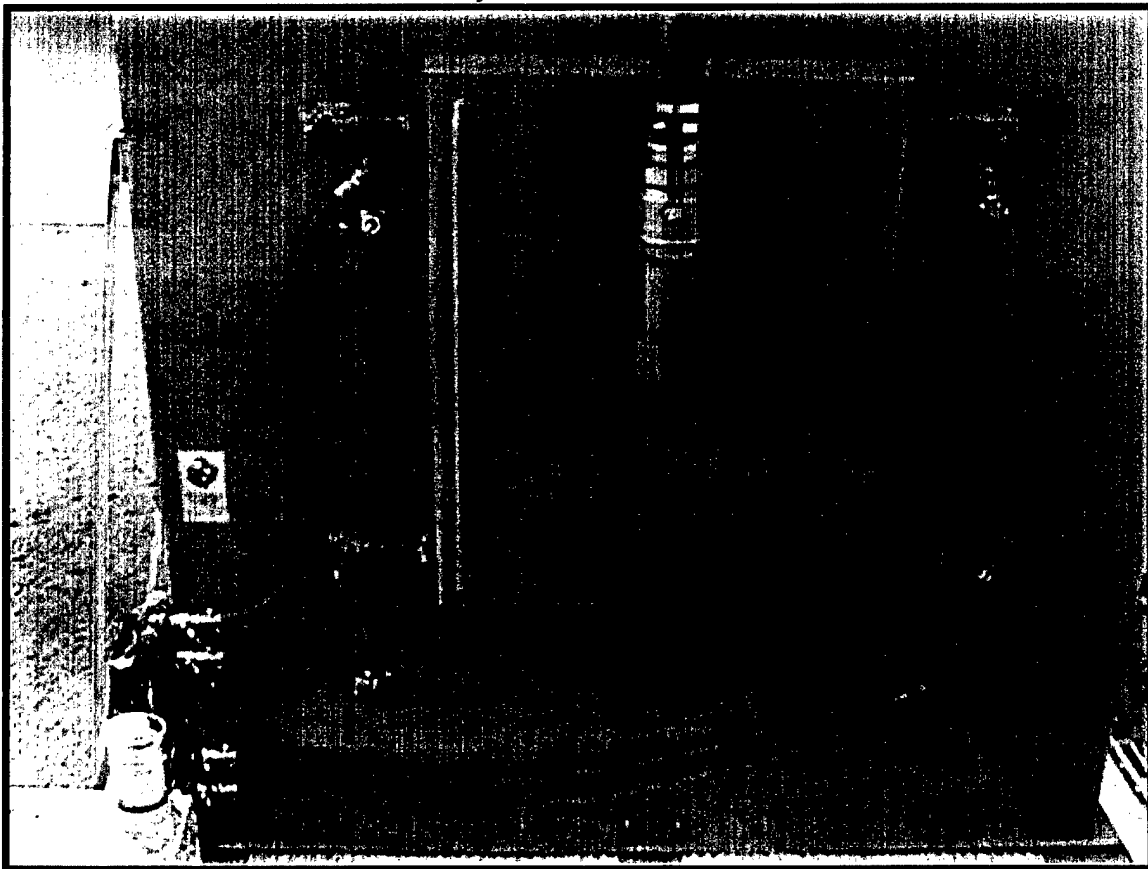
#### 4.5 Sampling Set-up at the Expansion Chamber



#### 4.6. Basic Components of the Donovan Blast Chamber



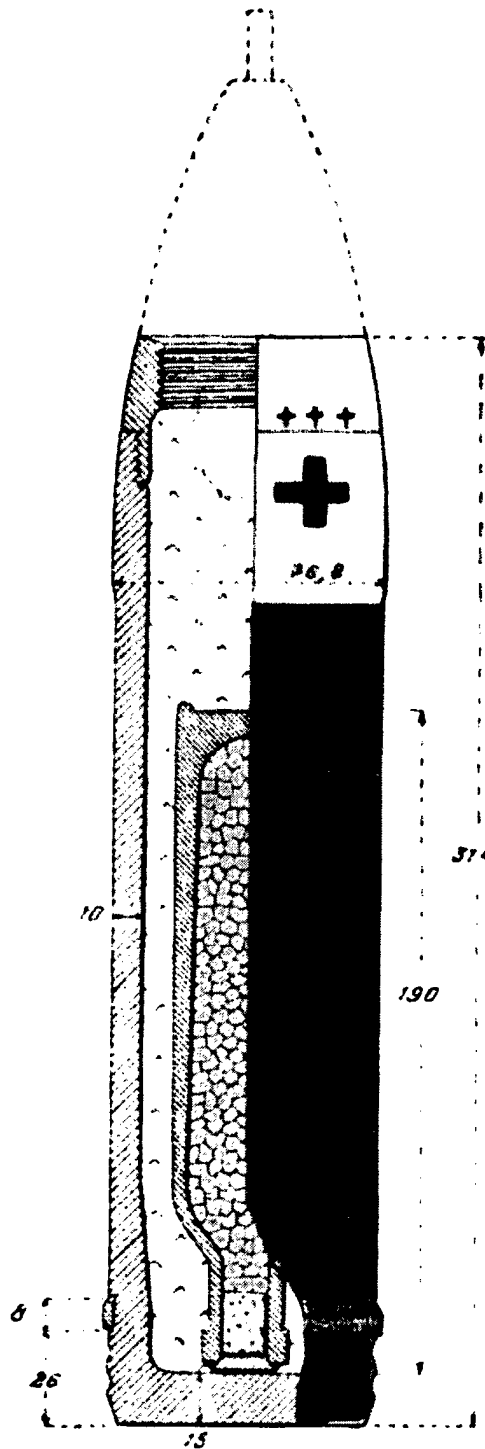
#### 4.7 Front View of Secondary Door of the T-10 Donovan Blast Chamber



#### 4.8 Wrapped Munition with Detonator Connected



4.9 7.7cm LFKGr Shell (GE) with Clark Fill, also known as Blaukreuz



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**APPENDIX 5.**

**QUALITY PROCESS SAMPLES**

**Quality Process Samples (QP) Results**

QP Identification Number	Amount Recovered (ng)	Amount Spiked (ng)	Percent Recovery (%)
0105160022-BEL	8.40	7.2	117
0105160023-BEL	7.39	7.2	103
0105170048-BEL	6.77	7.2	94
0105170049-BEL	5.57	7.2	77
0105180058-BEL	7.14	7.2	99
0105180059-BEL	7.13	7.2	99
0106140001-BEL	3.00	5.08	59
0106140002-BEL	2.74	5.08	54
0106200004-BEL	1.90	5.08	37
0106200005-BEL	1.80	5.08	35
0106210026-BEL	4.92	5.08	97
0106210027-BEL	5.84	5.08	115
0106270065-BEL	6.93	5.08	136
0106270066-BEL	7.54	5.08	148
0107020001-BEL	4.34	5.08	85
0107020002-BEL	3.79	5.08	75
0107030003-BEL	1.97	5.08	39
0107030004-BEL	2.89	5.08	57
0107040021-BEL	1.65	5.08	32
0107040022-BEL	1.72	5.08	45

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**APPENDIX 6.**  
**MINICAMS RESULTS**



Figure 6.1

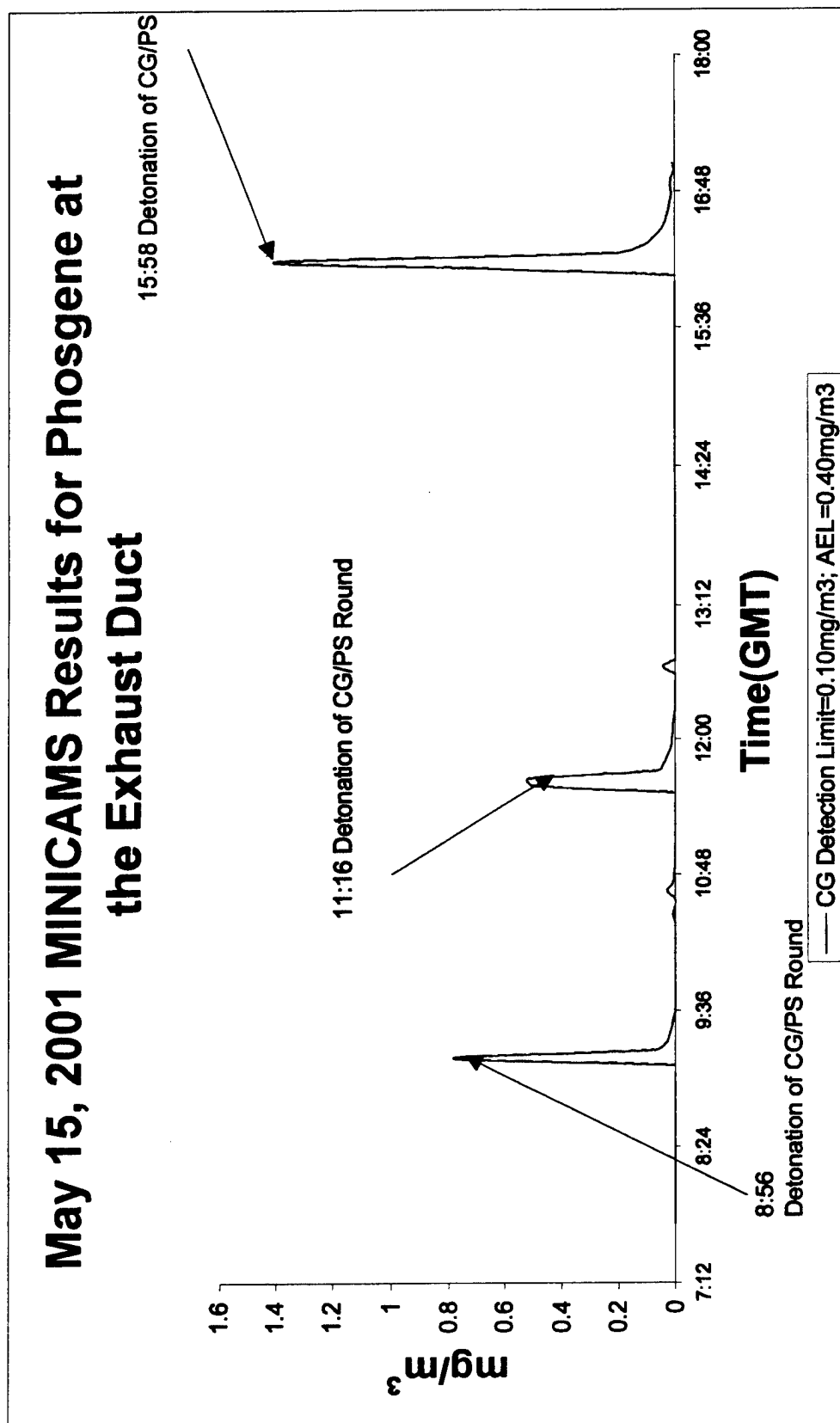


Figure 6.2

## May 15, 2001 MINICAMS Results for Chloropicrin at the Exhaust Duct

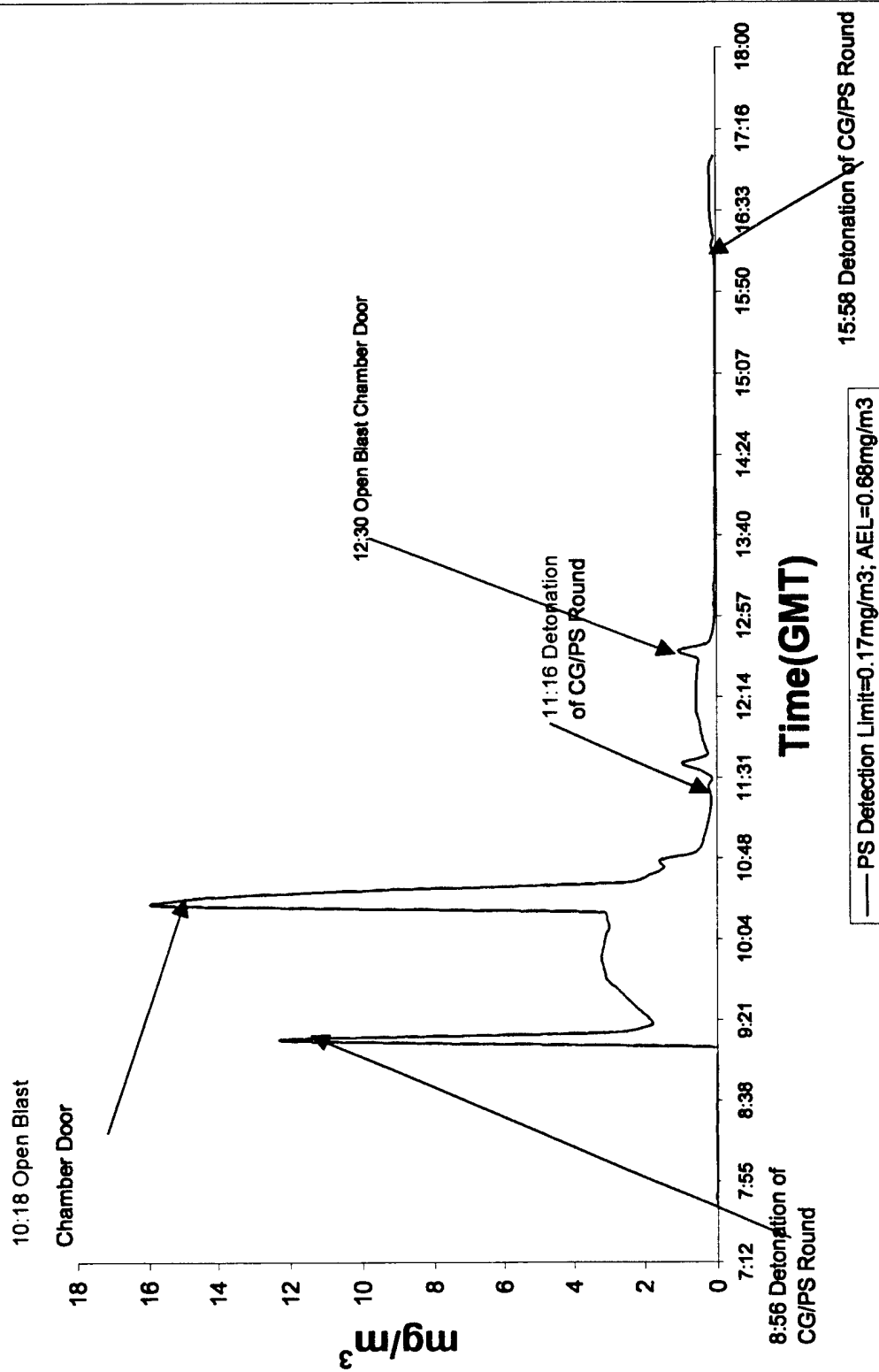


Figure 6.3

## May 15, 2001 MINICAMS Results for Chloropicrin at the Expansion Chamber

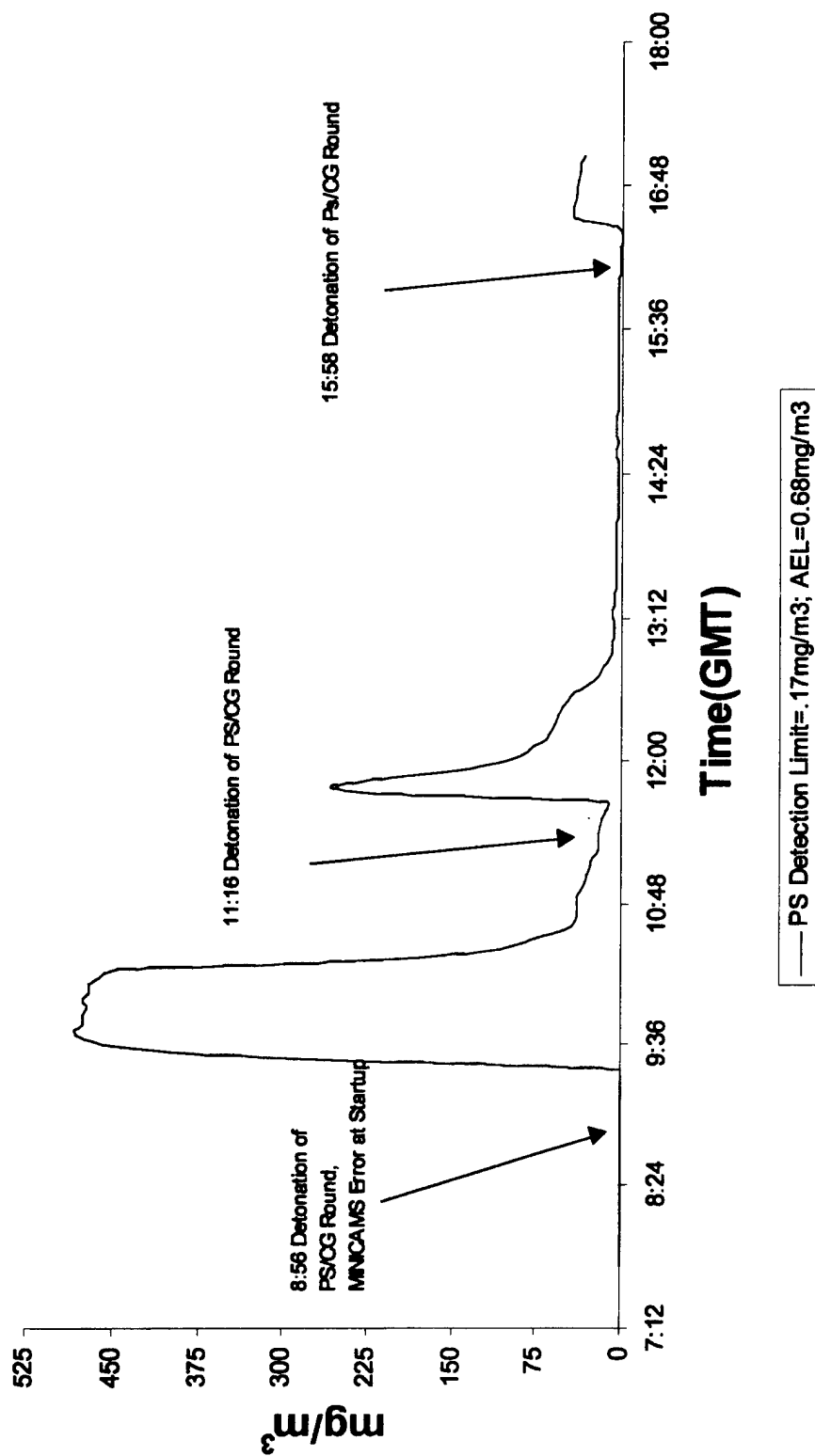


Figure 6.4

# **May 15, 2001 MINICAMS Results for Phosgene at the Expansion Chamber**

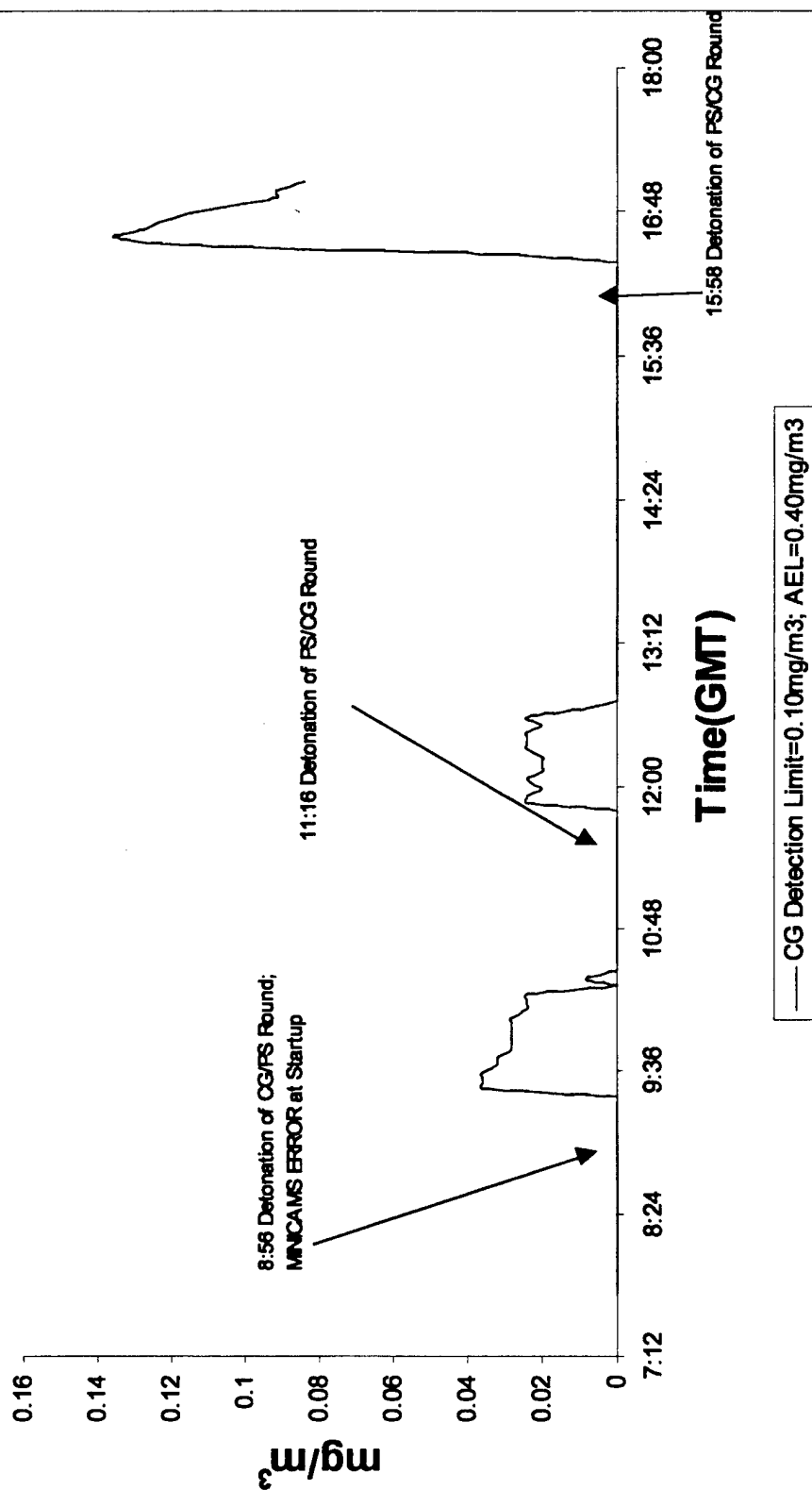


Figure 6.5

# May 16, 2001 MINICAMS Results for Mustard at the Exhaust Duct

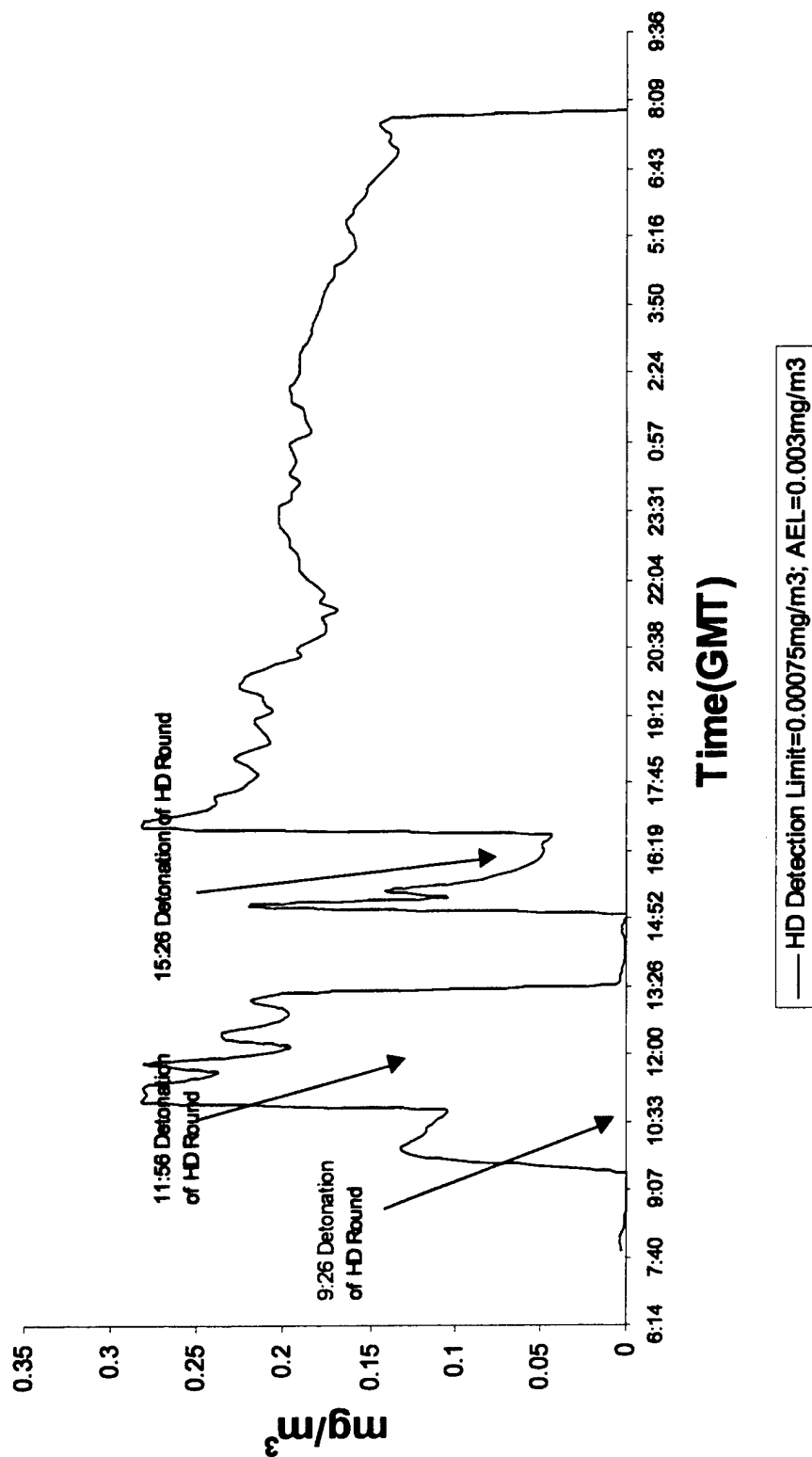


Figure 6.6

# **May 16, 2001 MINICAMS Results for Mustard at the Expansion Chamber**

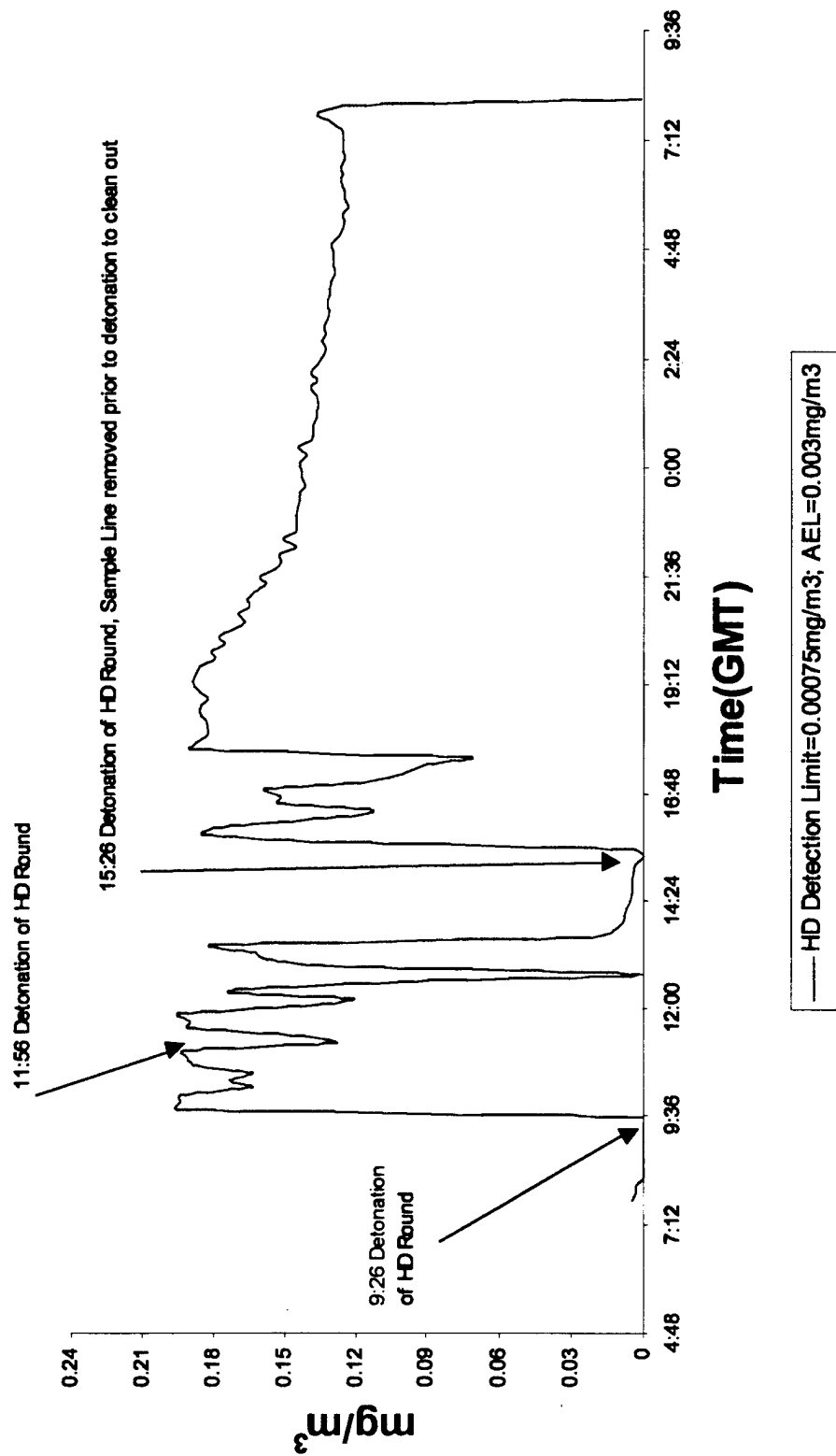


Figure 6.7

## May 17, 2001 MINICAMS Results for Mustard Residual in Exhaust Duct during DA Detonations

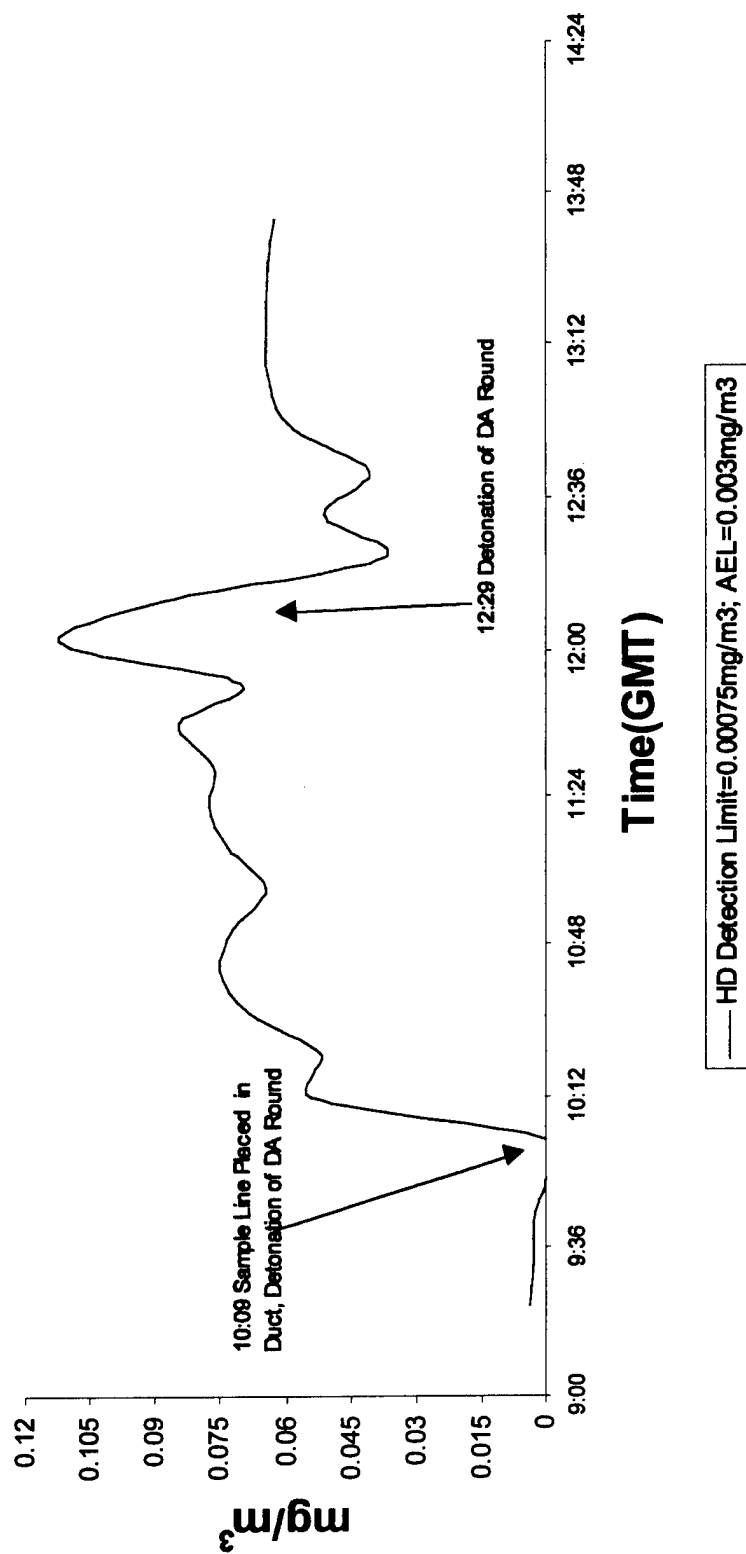


Figure 6.8

## May 17, 2001 MINICAMS Results for Mustard Residual during DA Detonations in the Expansion Chamber

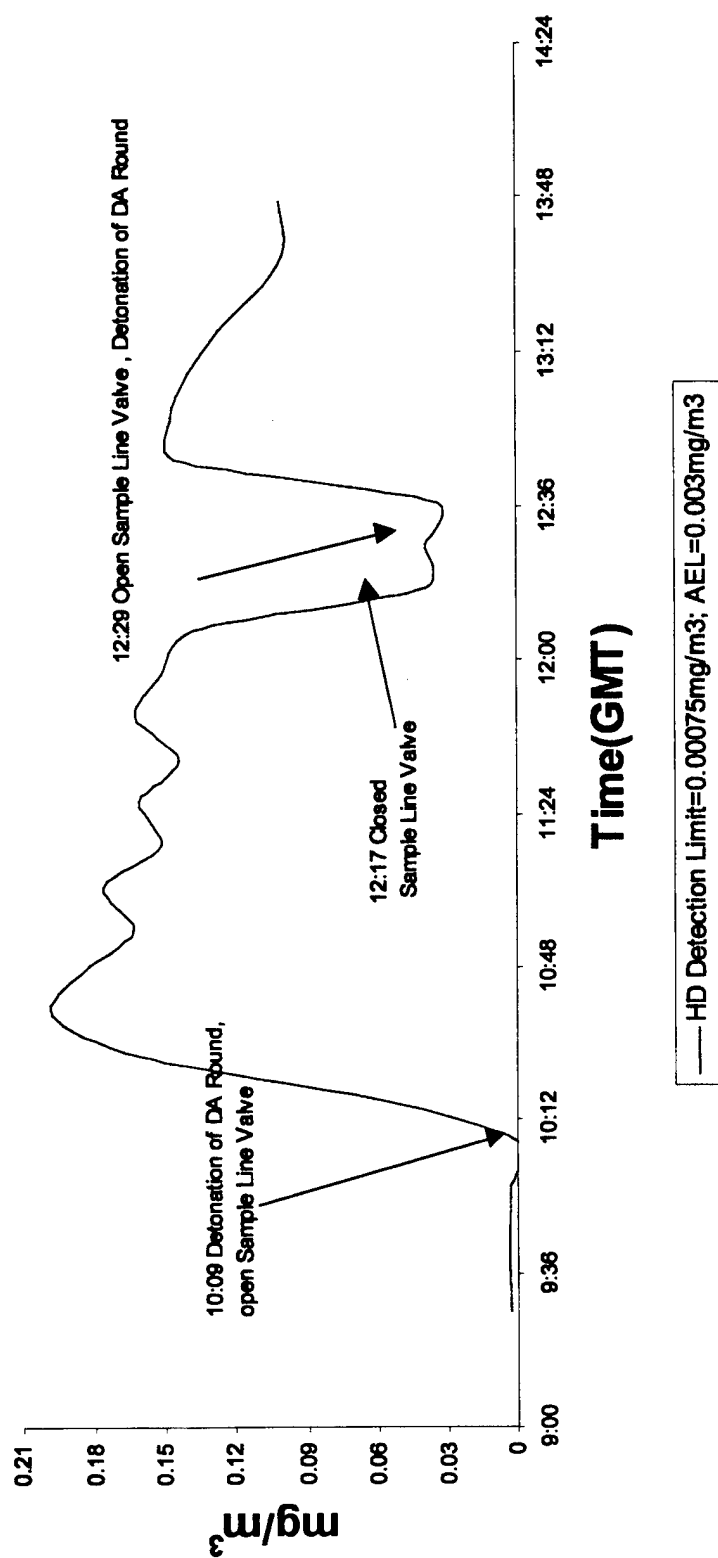




Figure 6.9

## May 18, 2001 MINICAMS Results For Chloropicrin at the Expansion Chamber

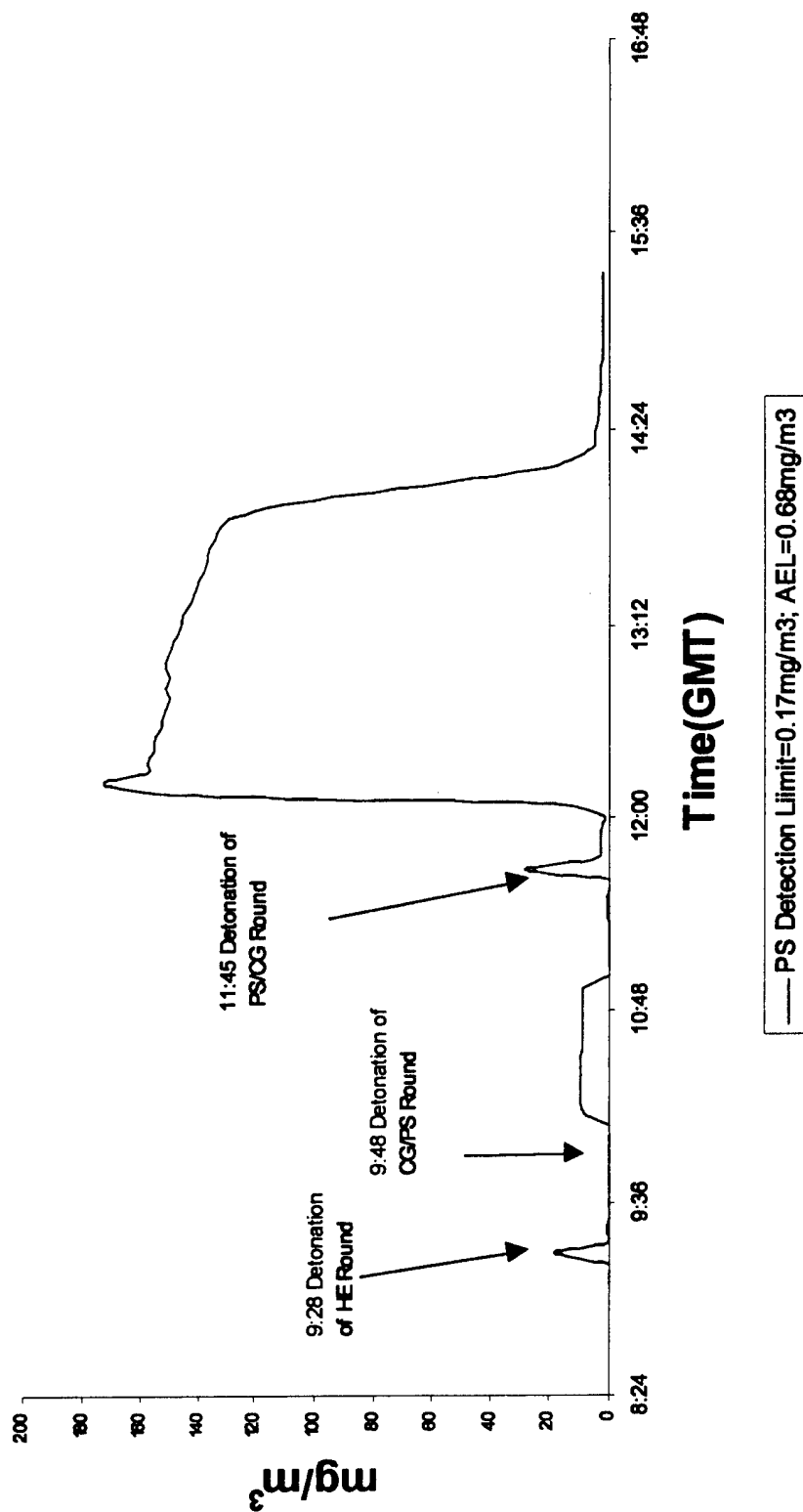


Figure 6.10

## May 18, 2001 MINICAMS Results for Chloropicrin at the Exhaust Duct

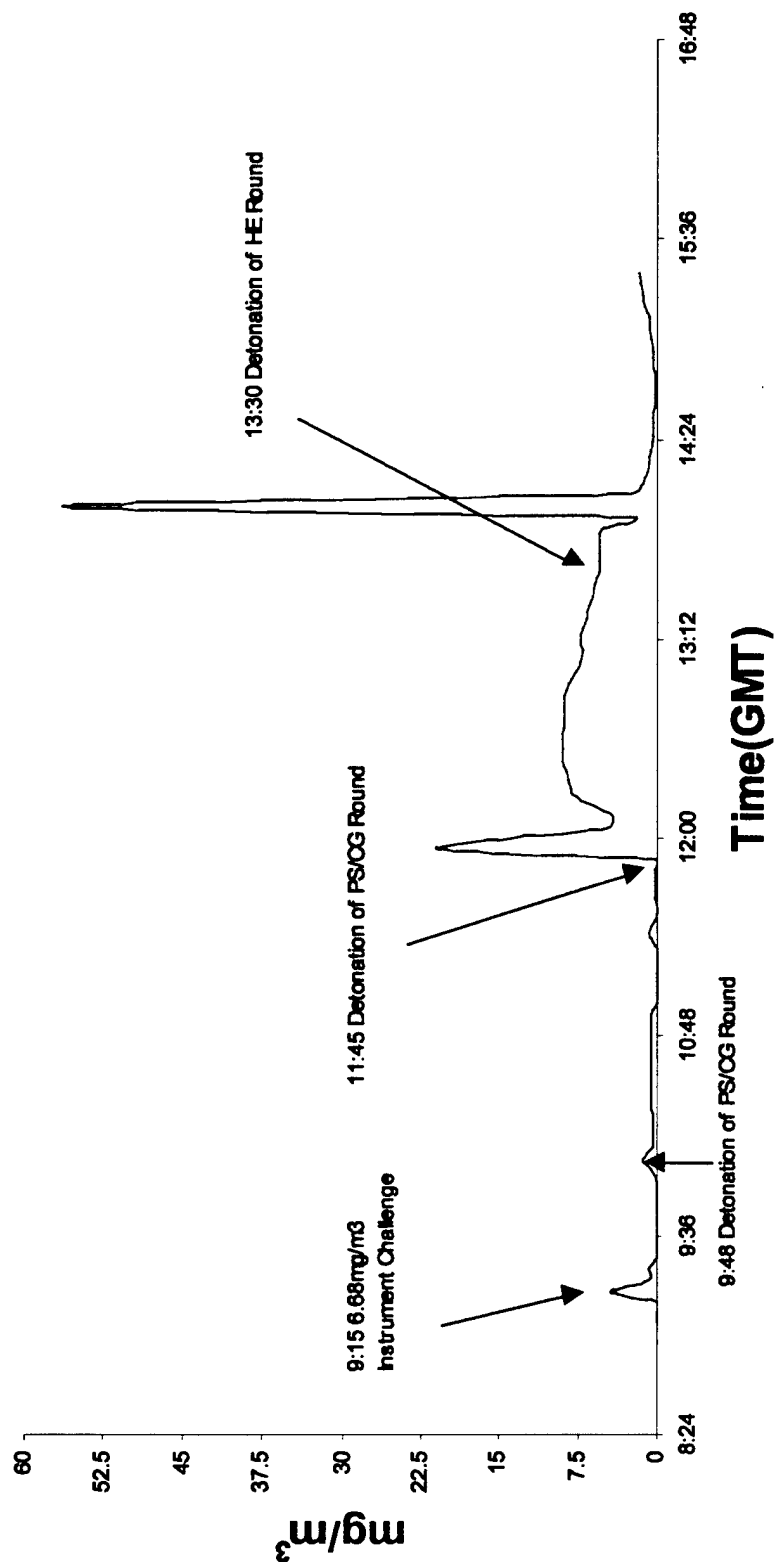


Figure 6.11

## May 21, 2001 MINICAMS Results for Mustard at the Exhaust Duct

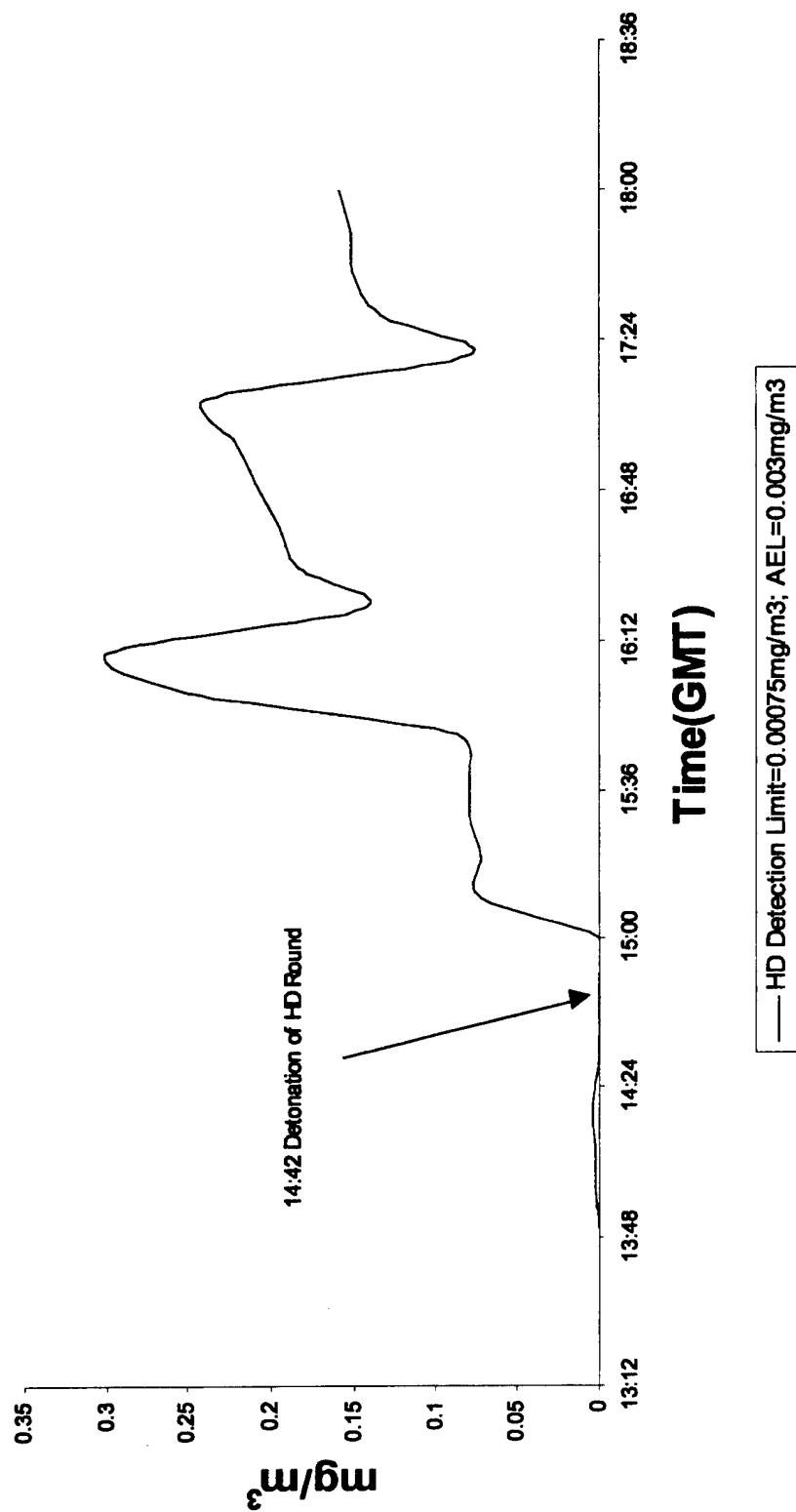


Figure 6.12

## May 21, 2001 MINICAMS Results for Mustard at the Expansion Chamber

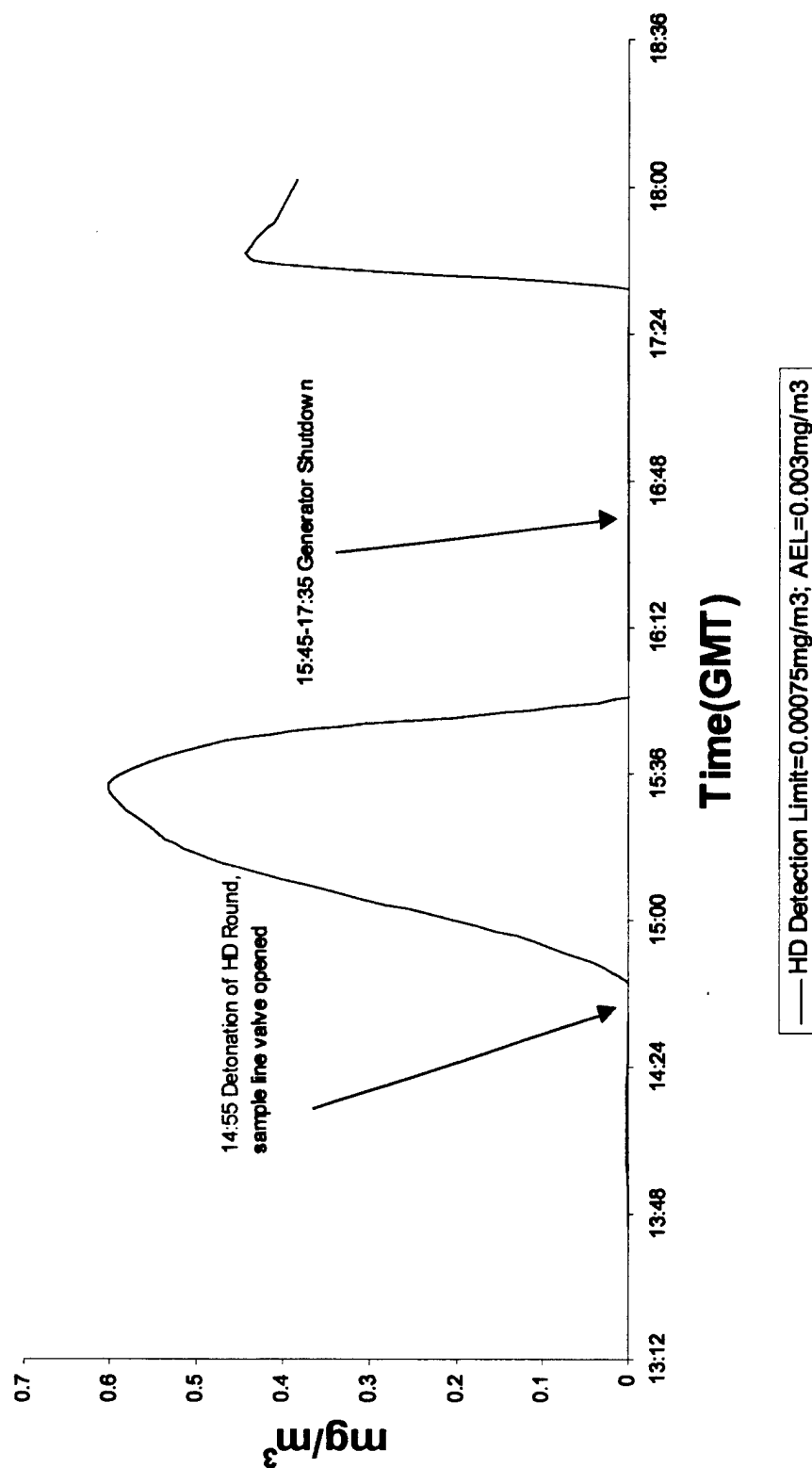


Figure 6.13

# June 20, 2001 MINICAMS Results for Mustard at the Exhaust Duct

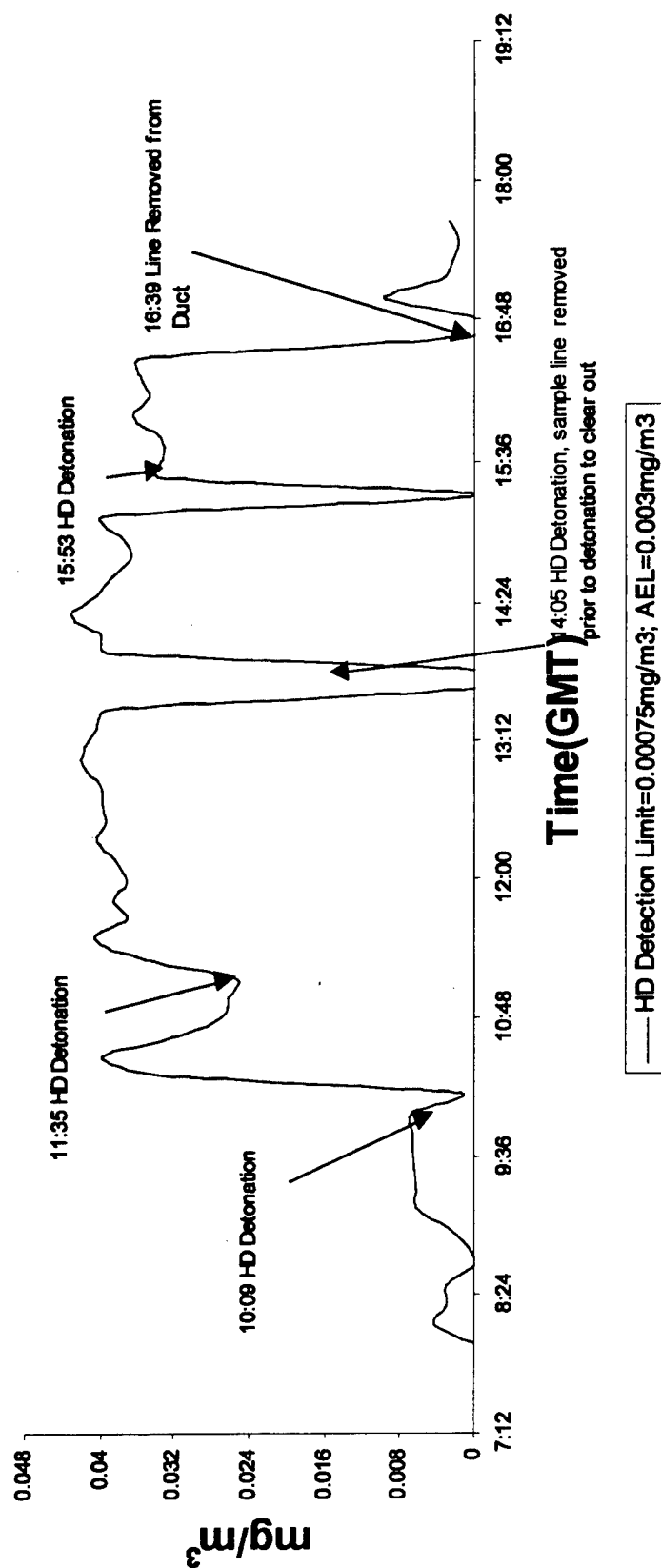


Figure 6.14

# June 20,2001 MINICAMS Results for Mustard at the Expansion Chamber

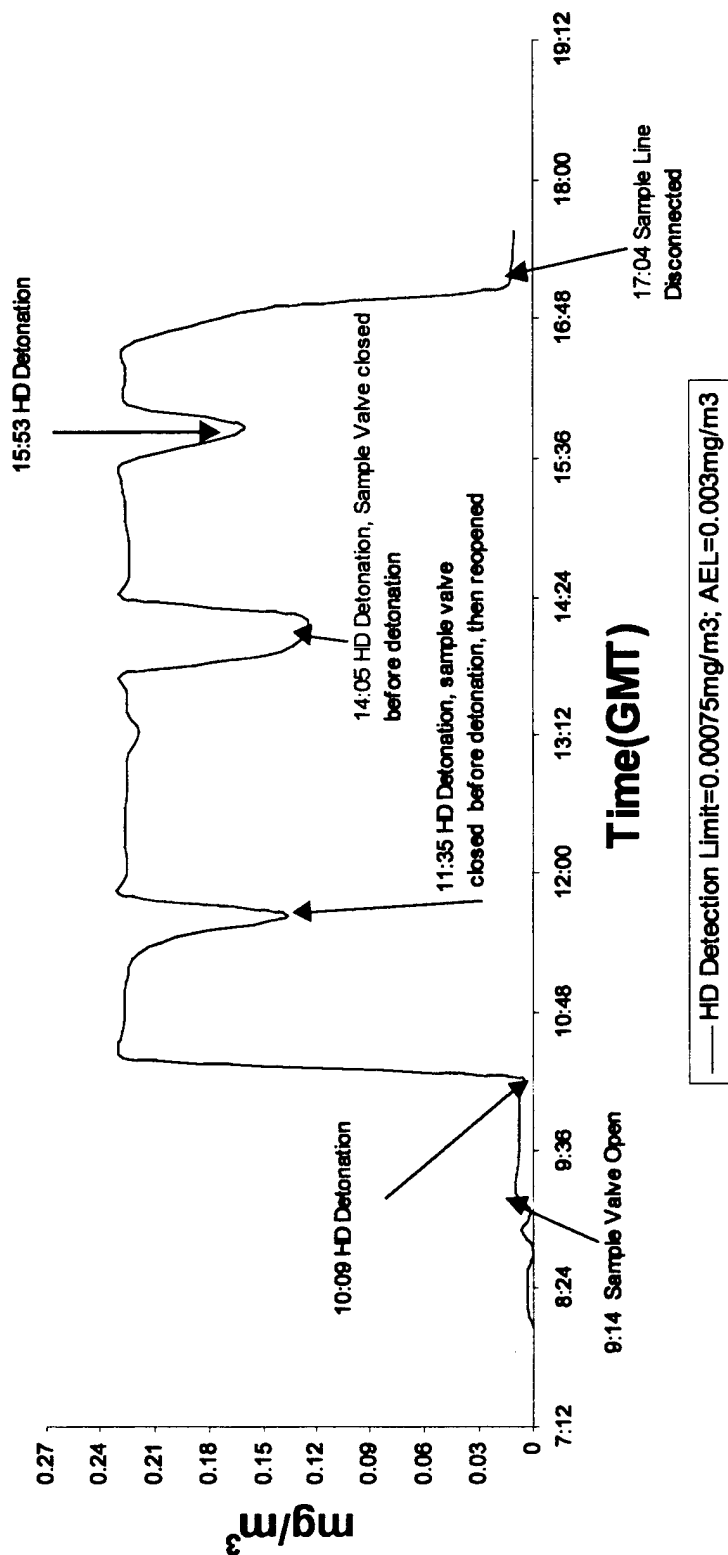


Figure 6.15

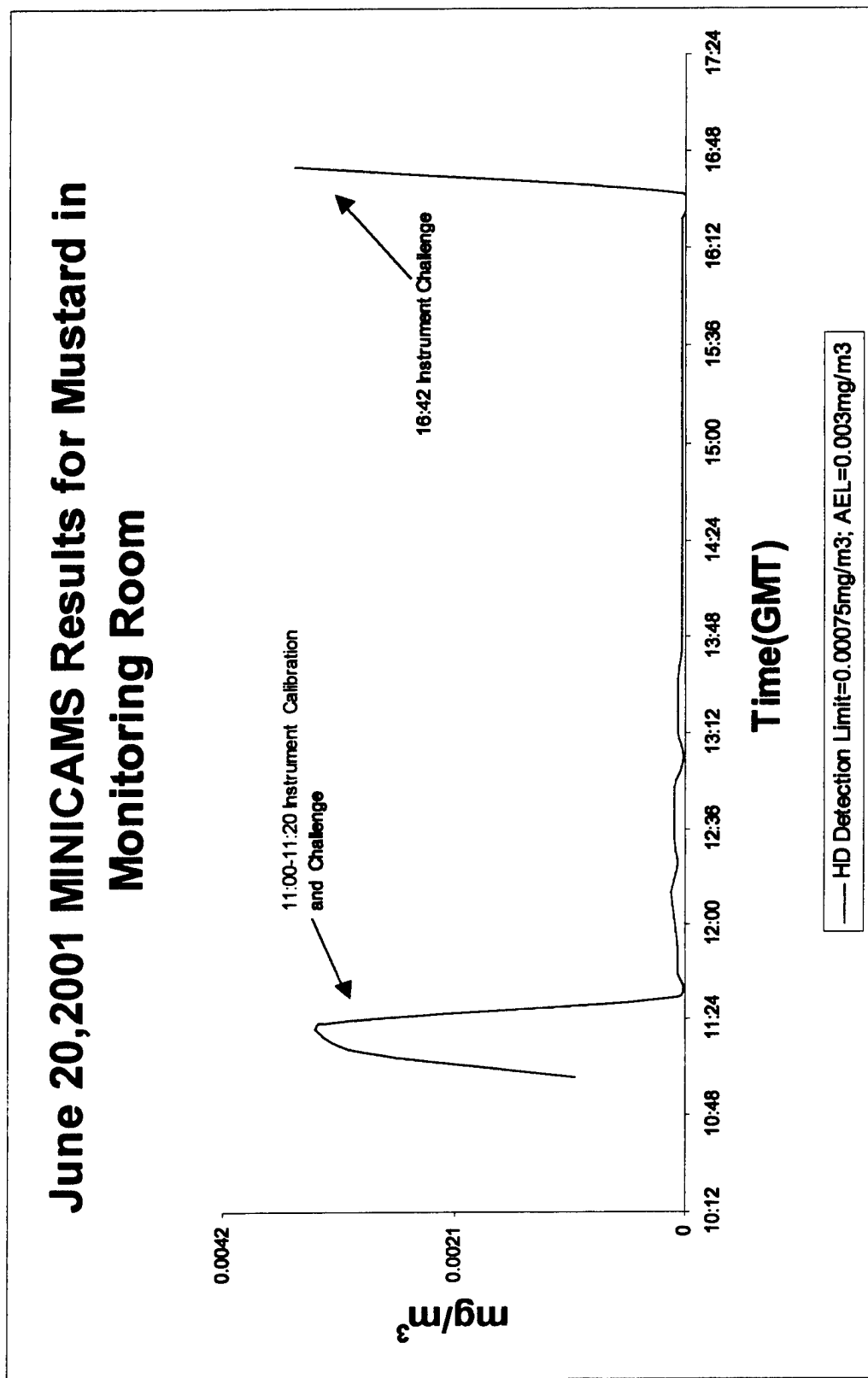


Figure 6.16

**June 21, 2001 MINICAMS Results for Mustard at  
the Exhaust Vent note: sample line  
contaminated, no breakthrough carbons**

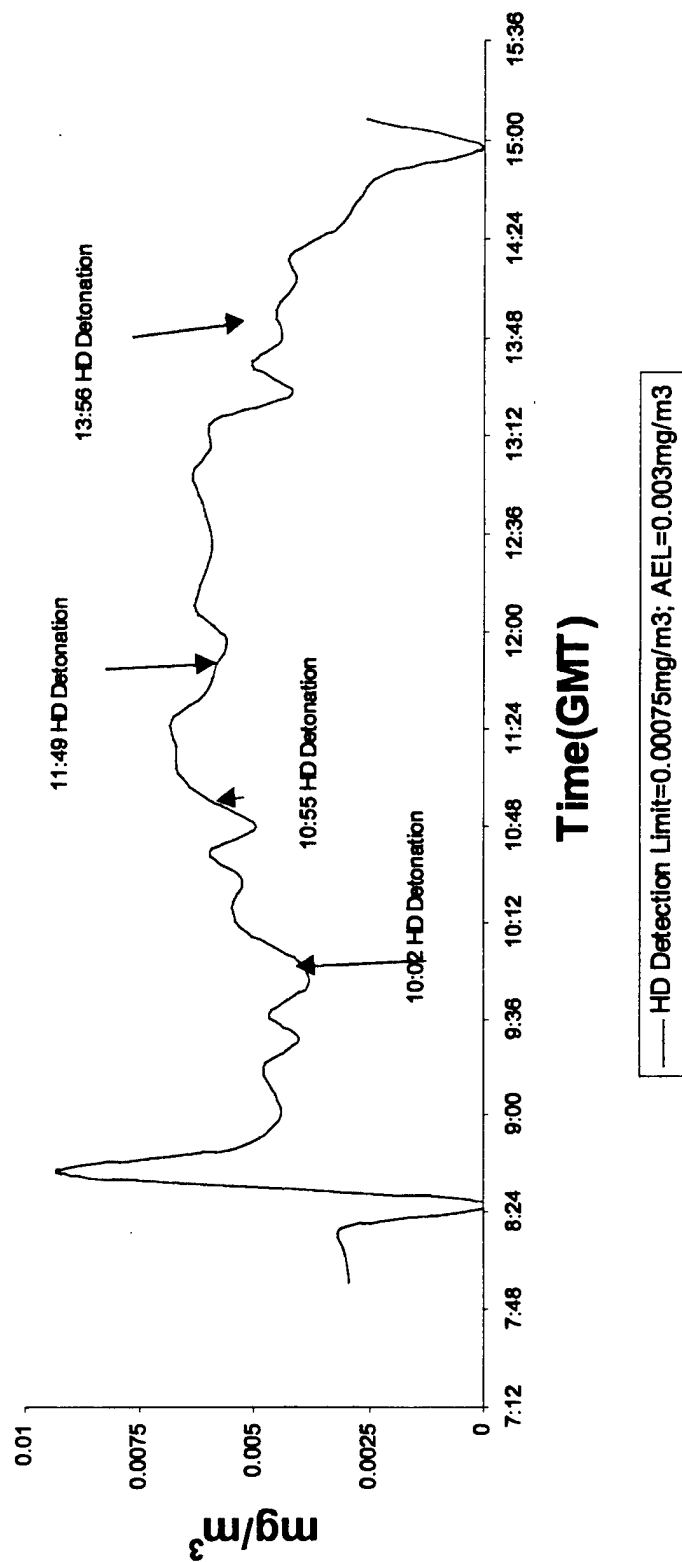




Figure 6.17

# June 21, 2001 MINICAMS Results for Mustard at the Expansion Chamber

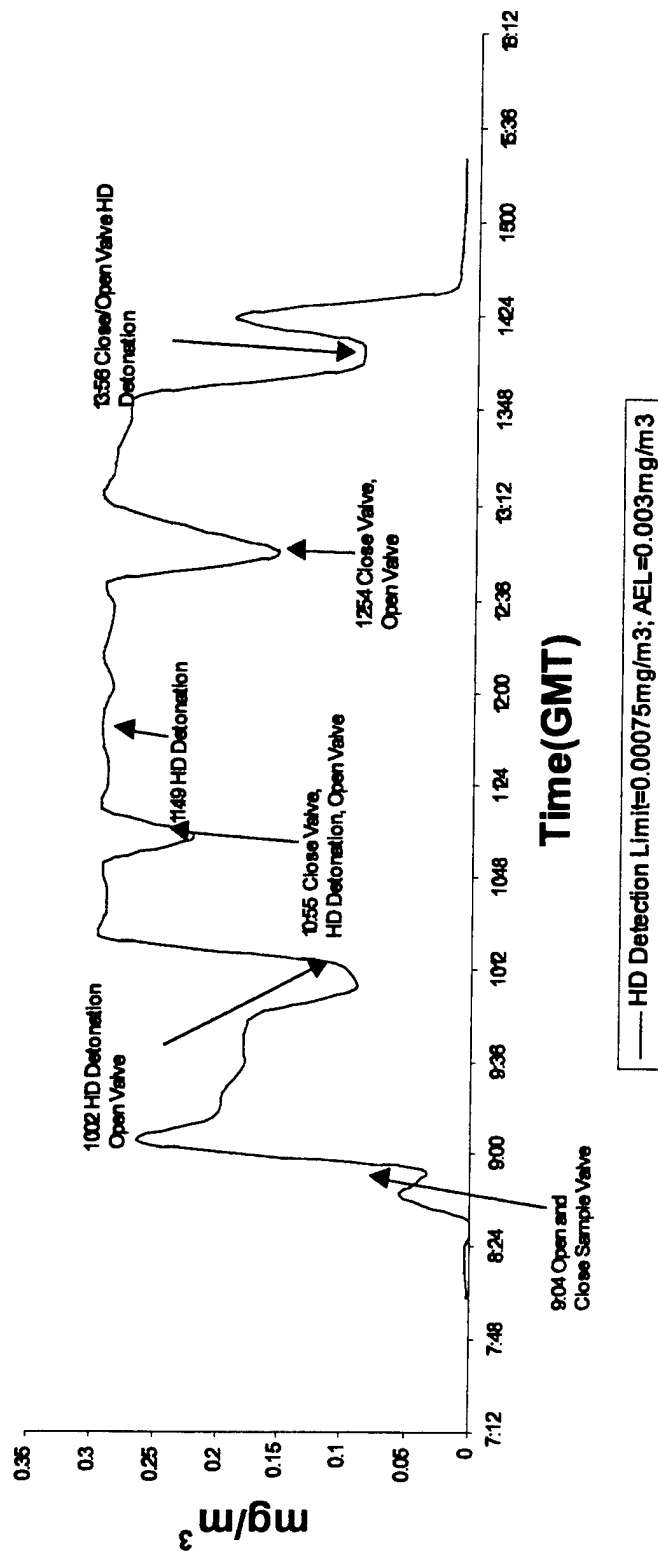
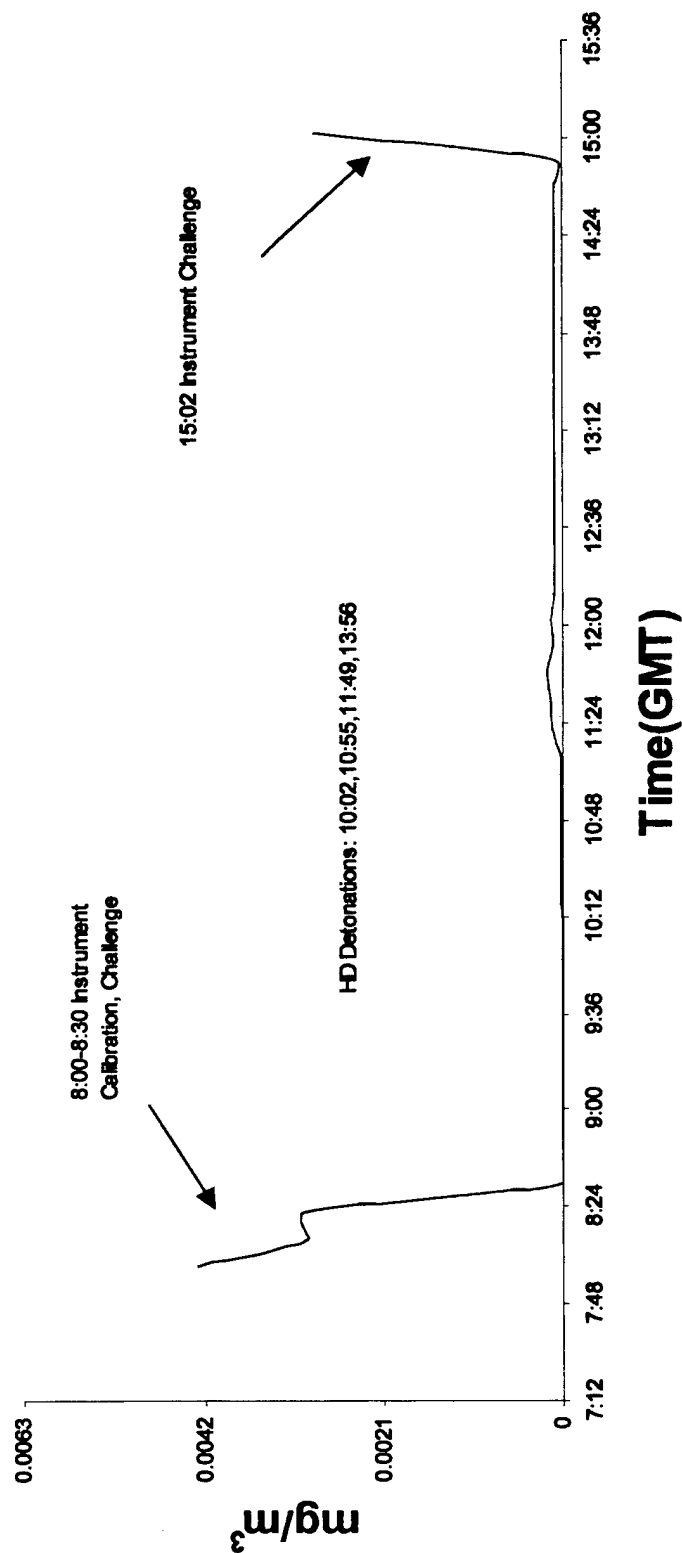


Figure 6.18

# June 21, 2001 MINICAMS Results for Mustard in Monitoring Room



— HD Detection Limit=0.00075mg/m3; AEL=0.003mg/m3

Figure 6.19

# June 22, 2001 MINICAMS Results for Mustard at the Exhaust Vent

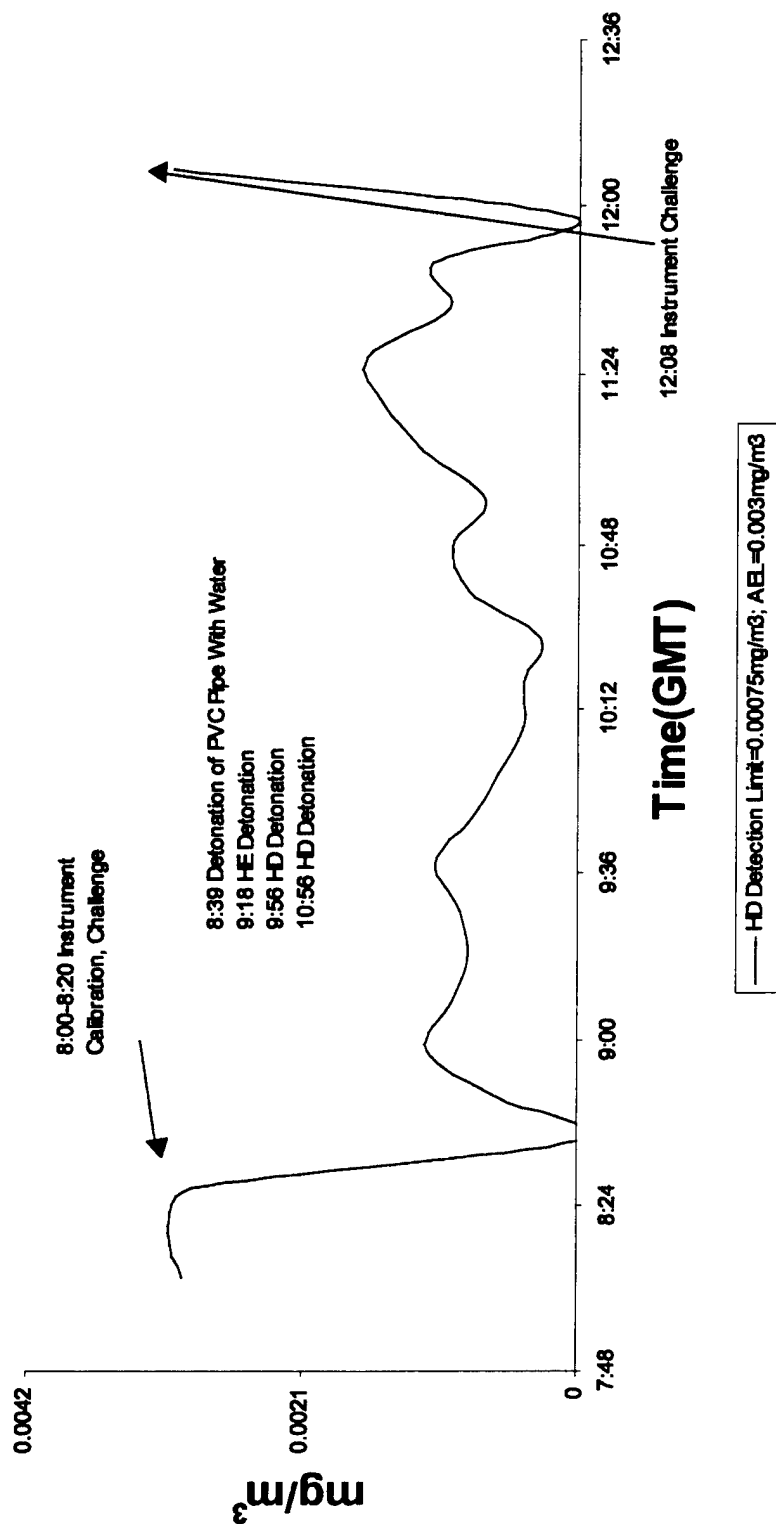
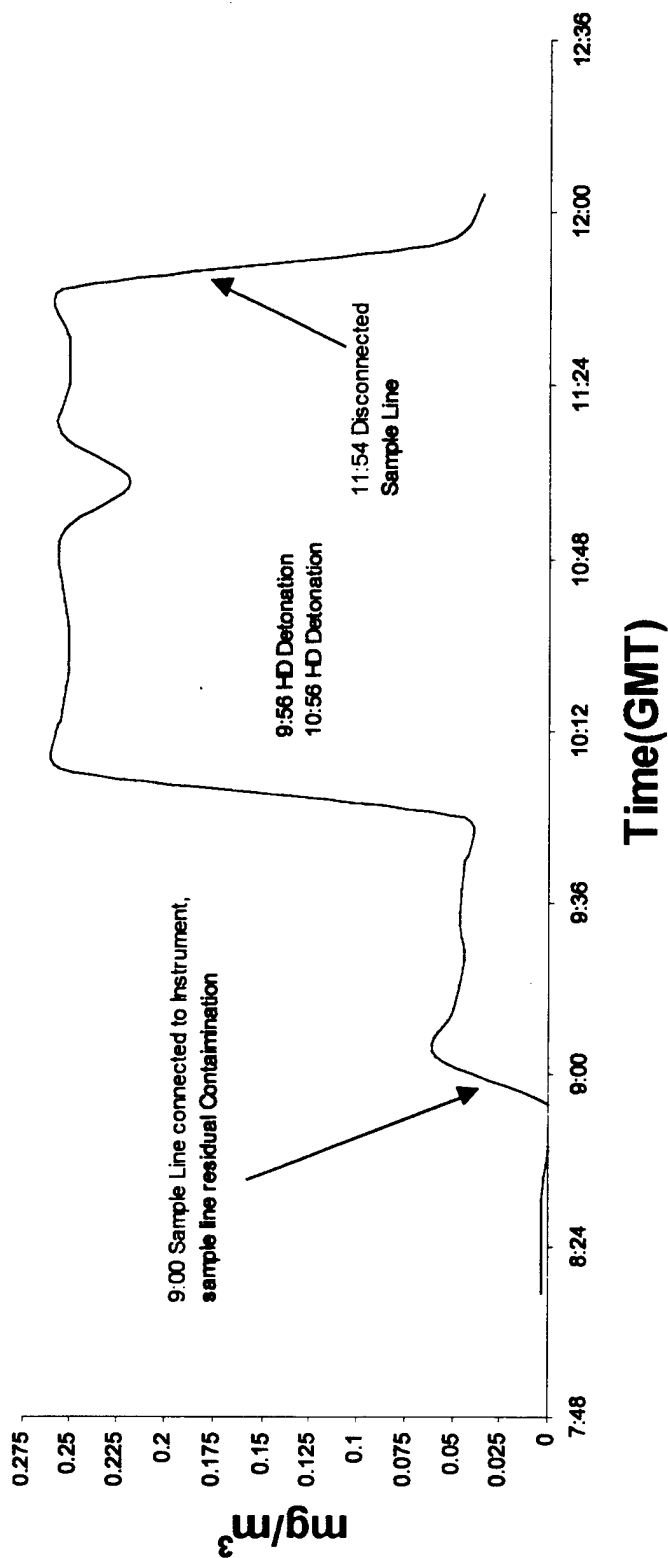


Figure 6.20

# June 22,2001 MINICAMS Results for Mustard at the Expansion Chamber



— HD Detection Limit=0.00075mg/m<sup>3</sup>; AEL=0.003mg/m<sup>3</sup>

Figure 6.21

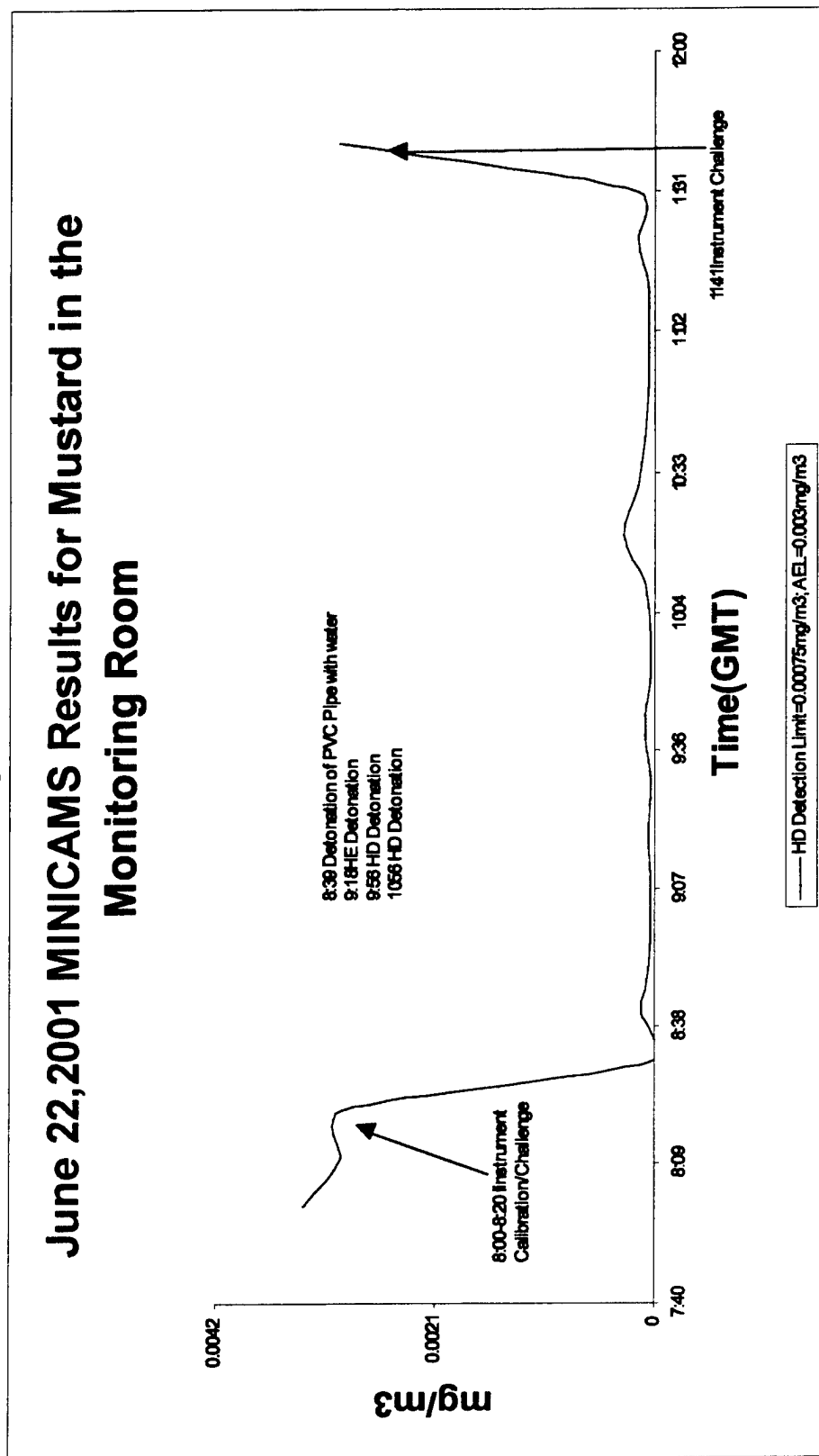


Figure 6.22

# June 27, 2001 MINICAMS Results for Phosgene at the Exhaust Vent

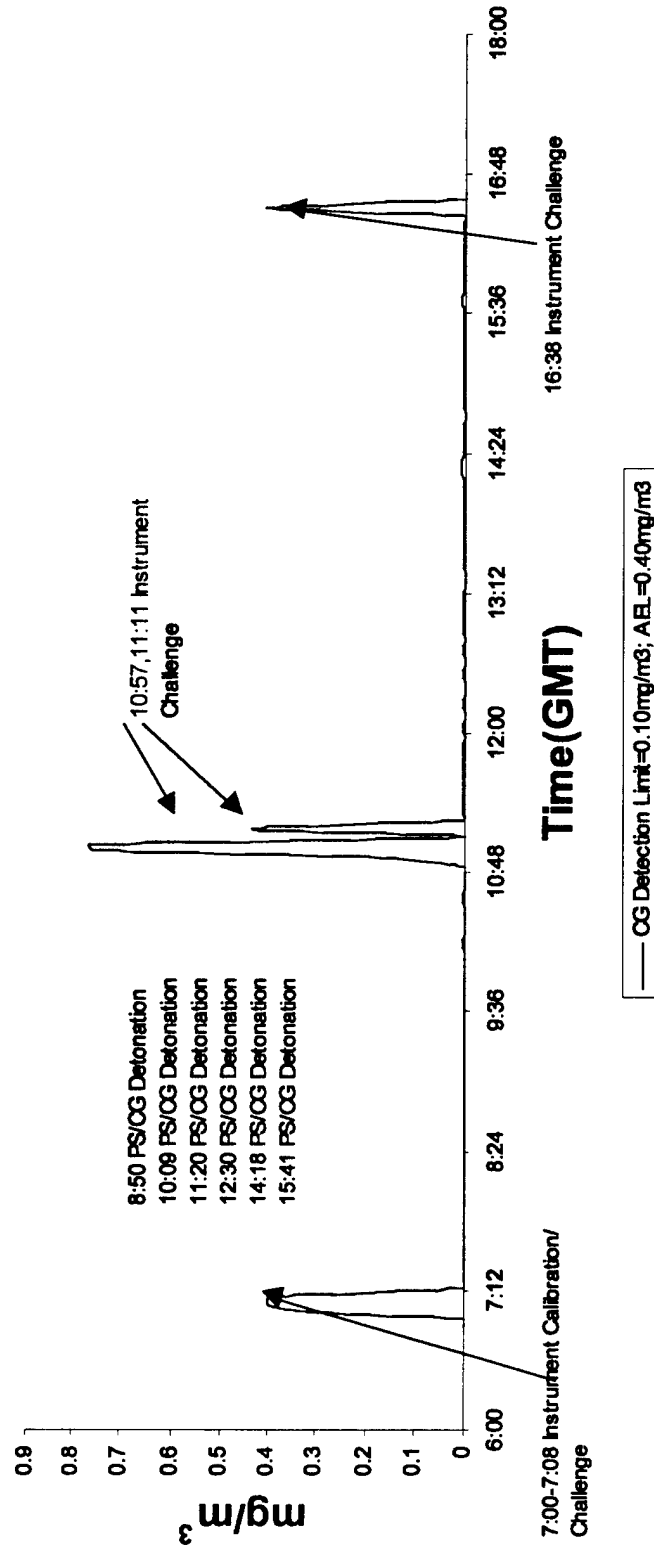


Figure 6.23

# June 27, 2001 MINICAMS Results for Chloropicrin at the Exhaust Vent

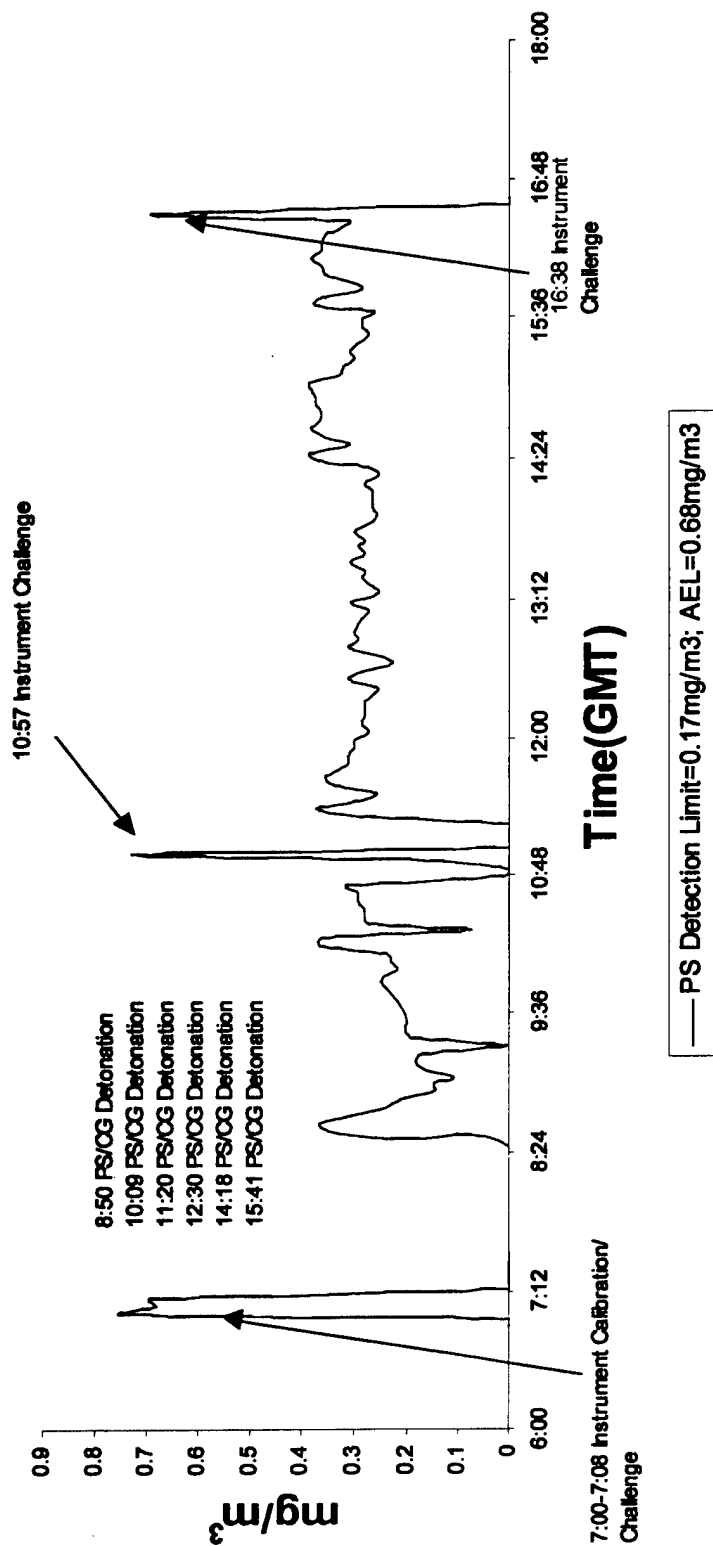


Figure 6.24

## June 27, 2001 MINICAMS Results for Phosgene at the Expansion Chamber

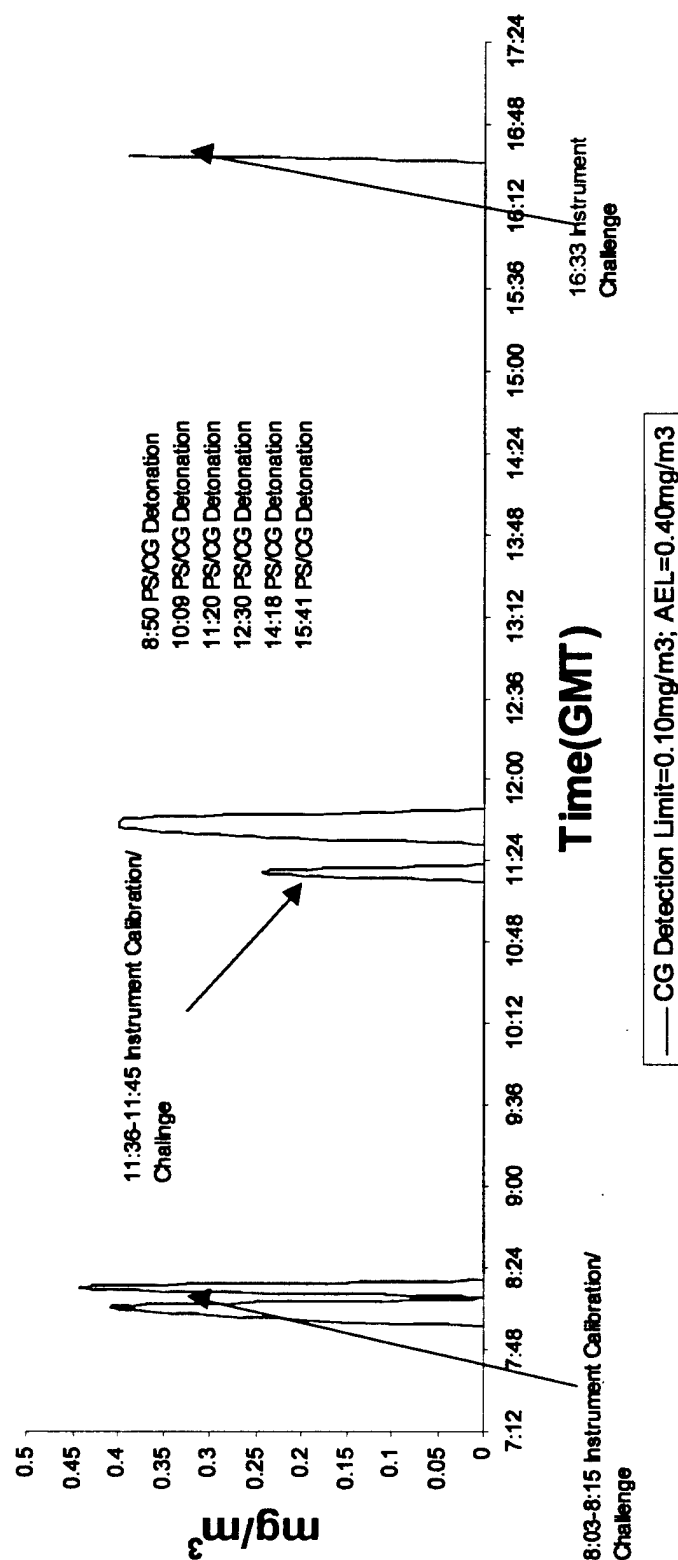




Figure 6.25

# July 27, 2001 MINICAMS Results for Chloropicrin at the Expansion Chamber

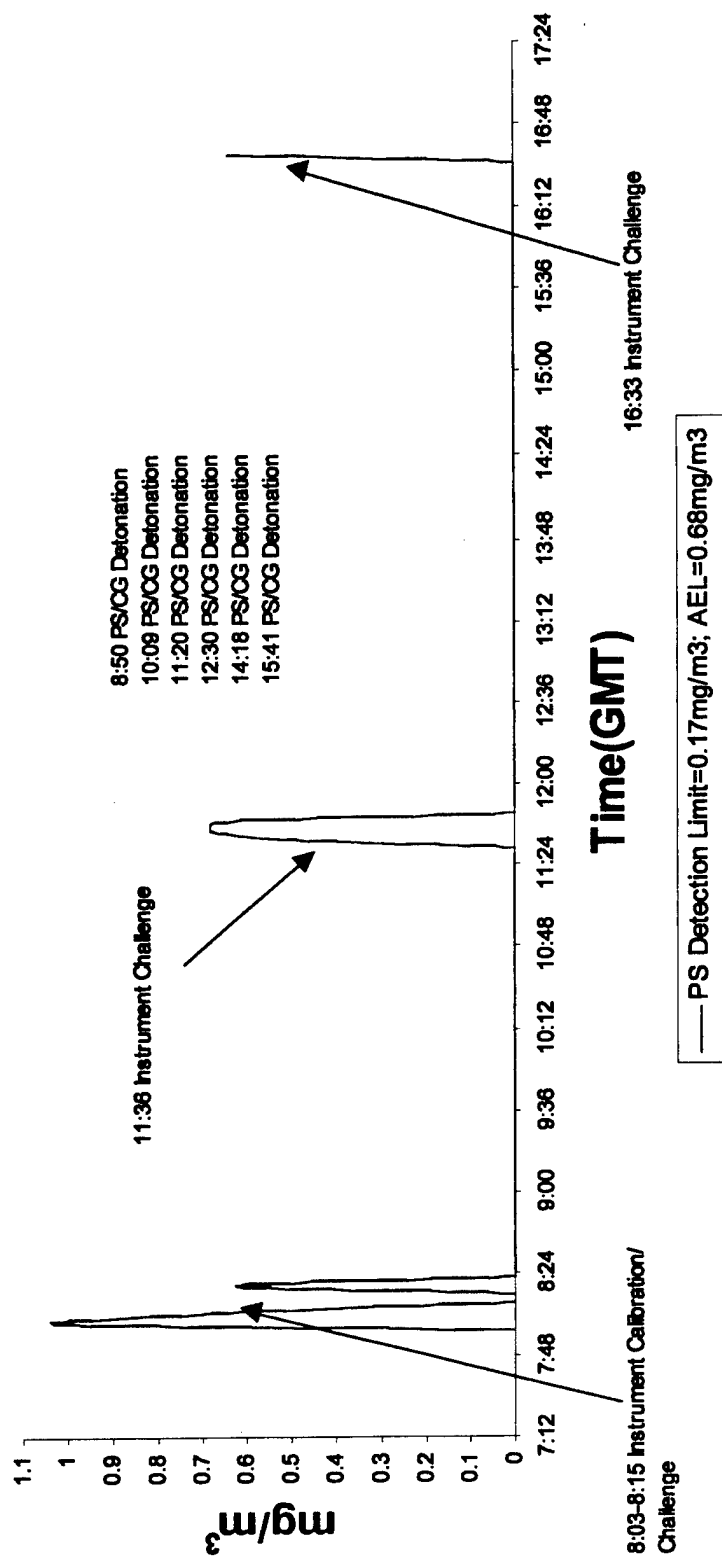


Figure 6.26

# June 28, 2001 MINICAMS Results for Phosgene at the Exhaust Vent

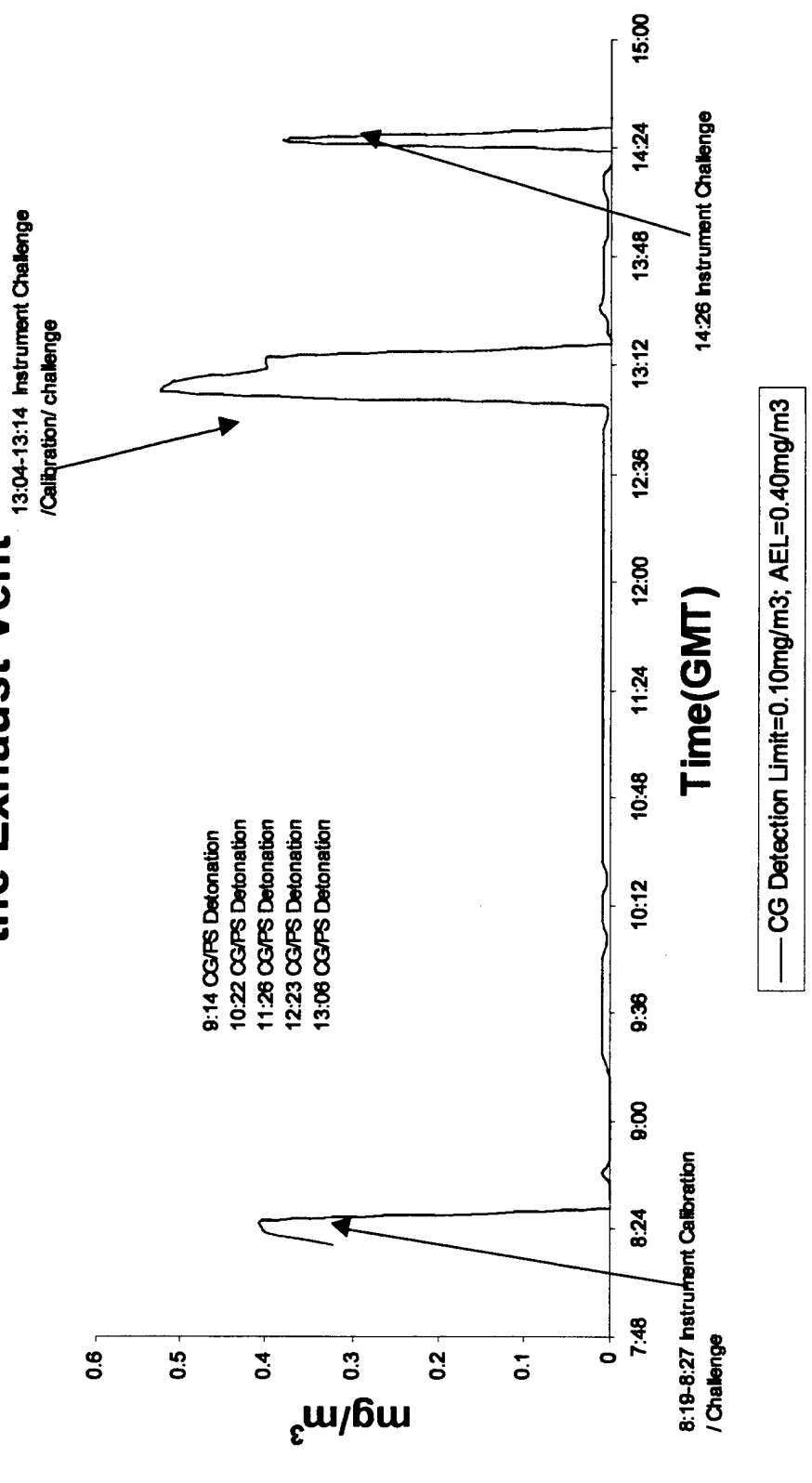


Figure 6.27

# June 28, 2001 MINICAMS Results for Chloropicrin at the Exhaust Vent

14:26 Disconnect Sample Line  
Challenge Instrument

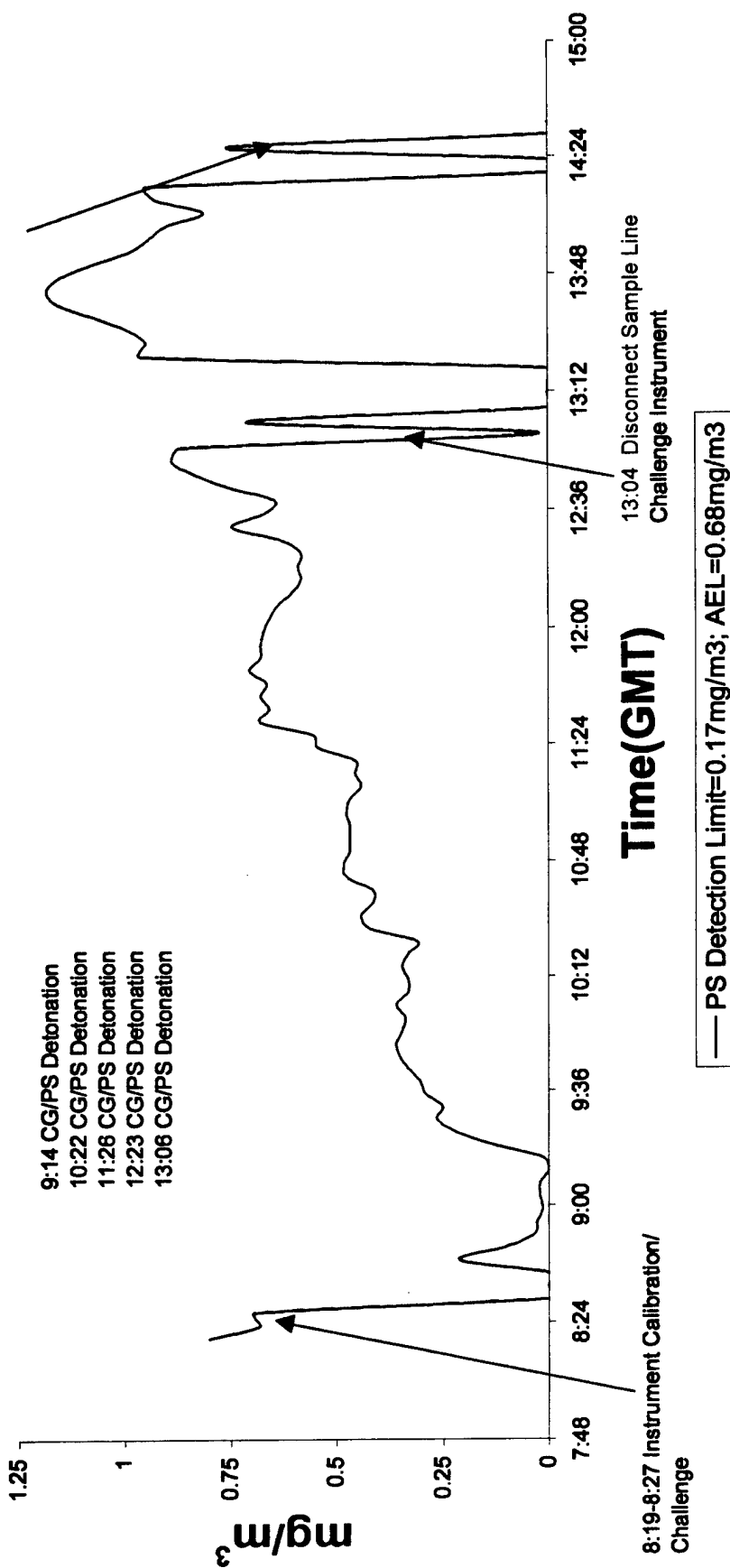


Figure 6.28

# June 28, 2001 MINICAMS Results for Phosgene at the Expansion Chamber

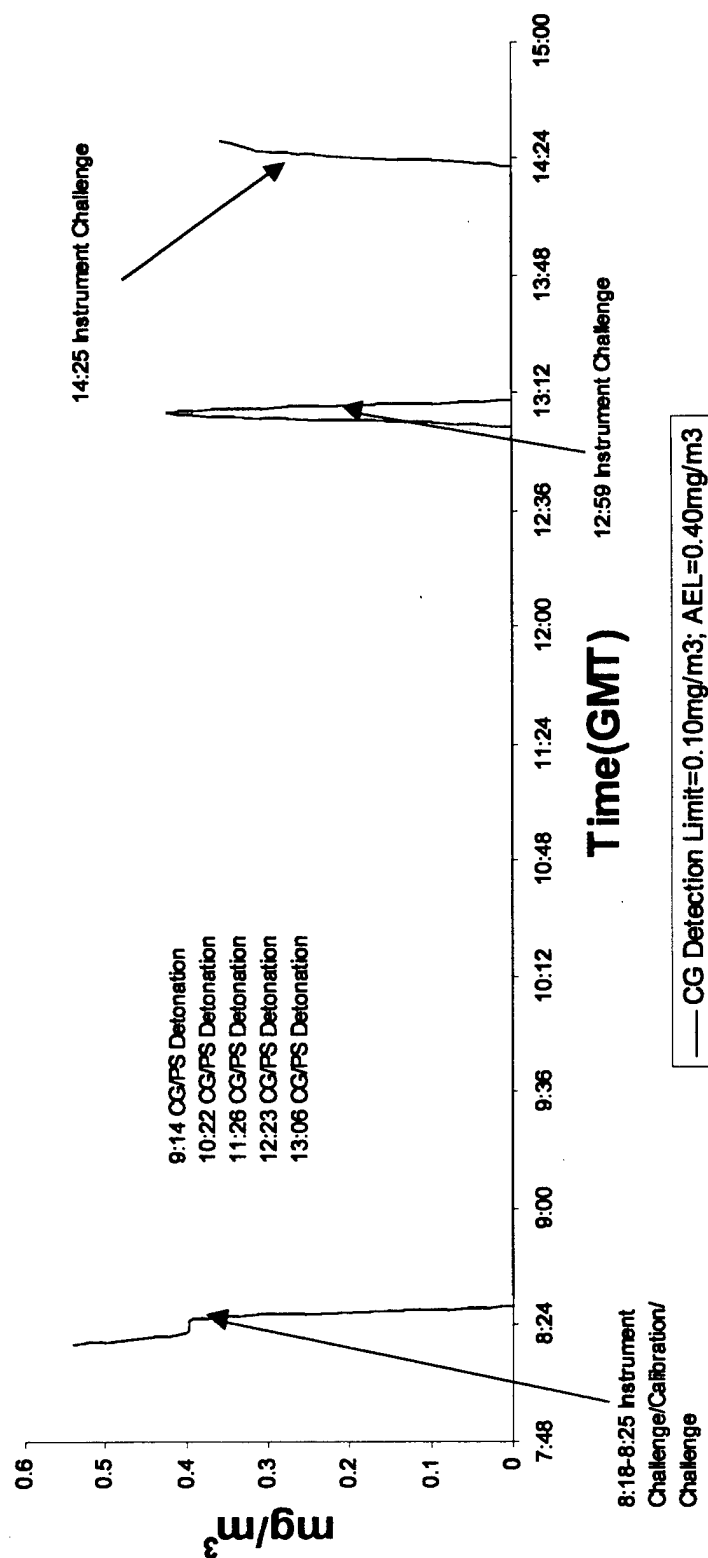


Figure 6.29

# June 28, 2001 MINICAMS Results for Chloropicrin at the Expansion Chamber

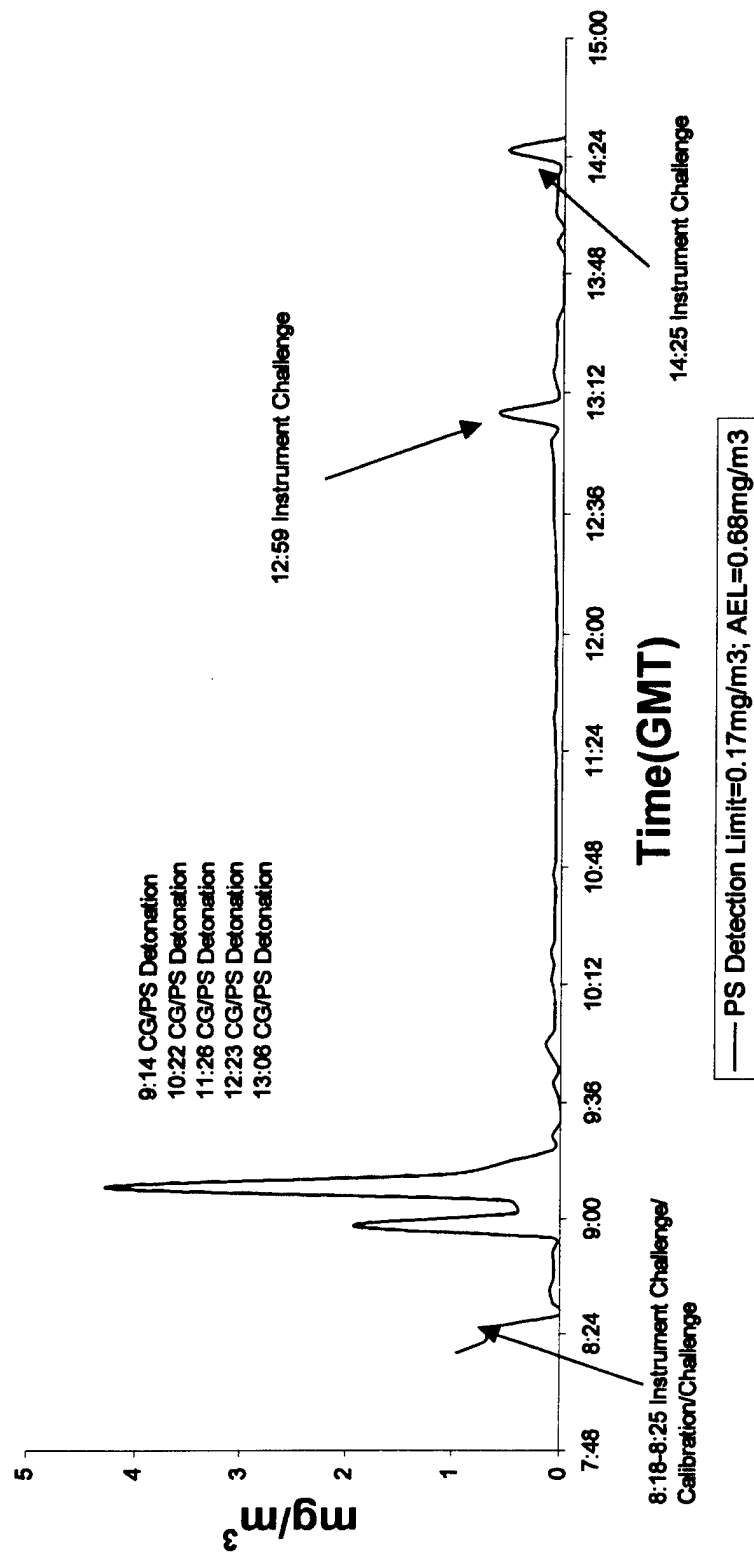


Figure 6.30

# June 29, 2001 MINICAMS Results for Phosgene at the Exhaust Vent



Figure 6.31

# June 29, 2001 MINICAMS Results for Chloropicrin at the Exhaust Vent

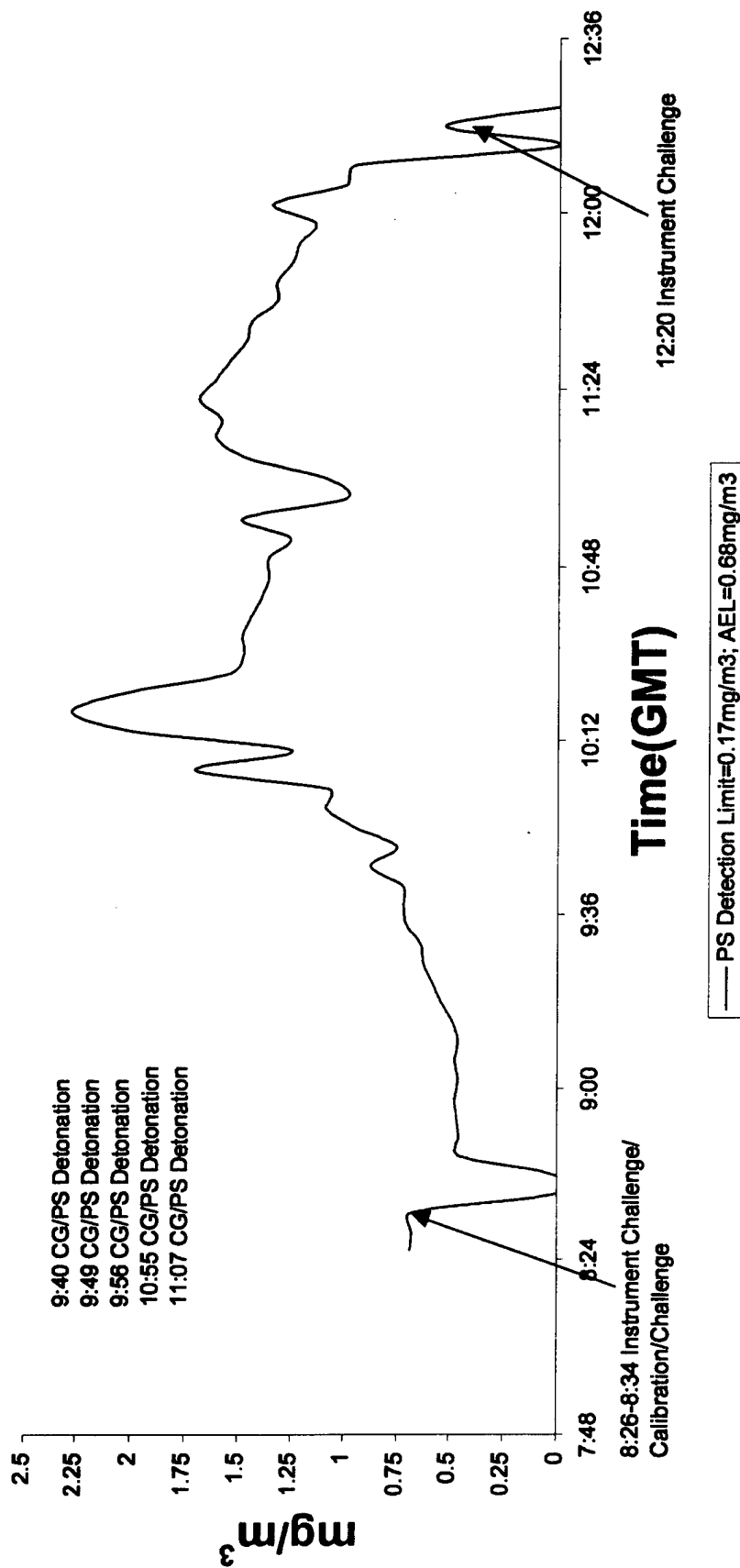


Figure 6.32

# June 29, 2001 MINICAMS Results for Phosgene at the Expansion Chamber

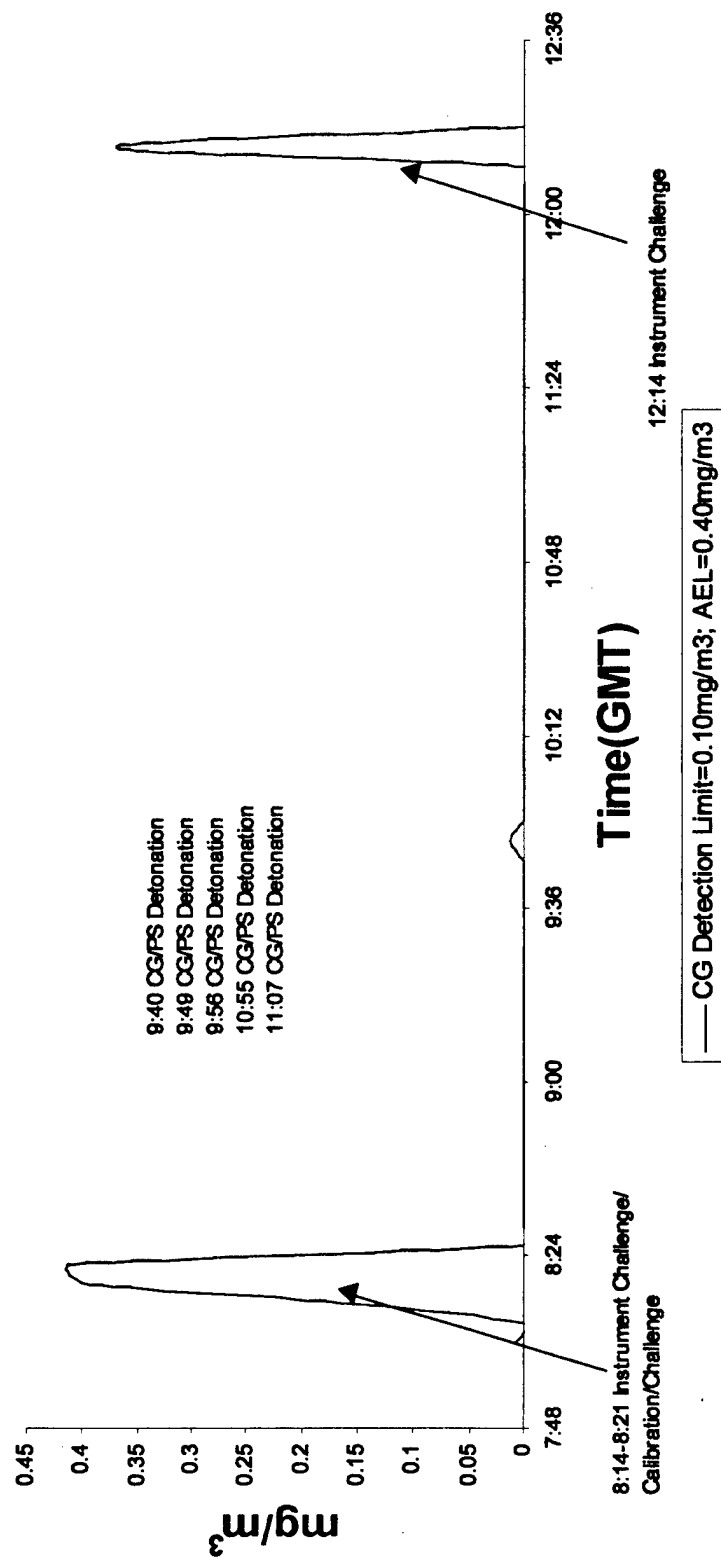




Figure 6.33

# June 29, 2001 MINICAMS Results for Chloropicrin at the Expansion Chamber

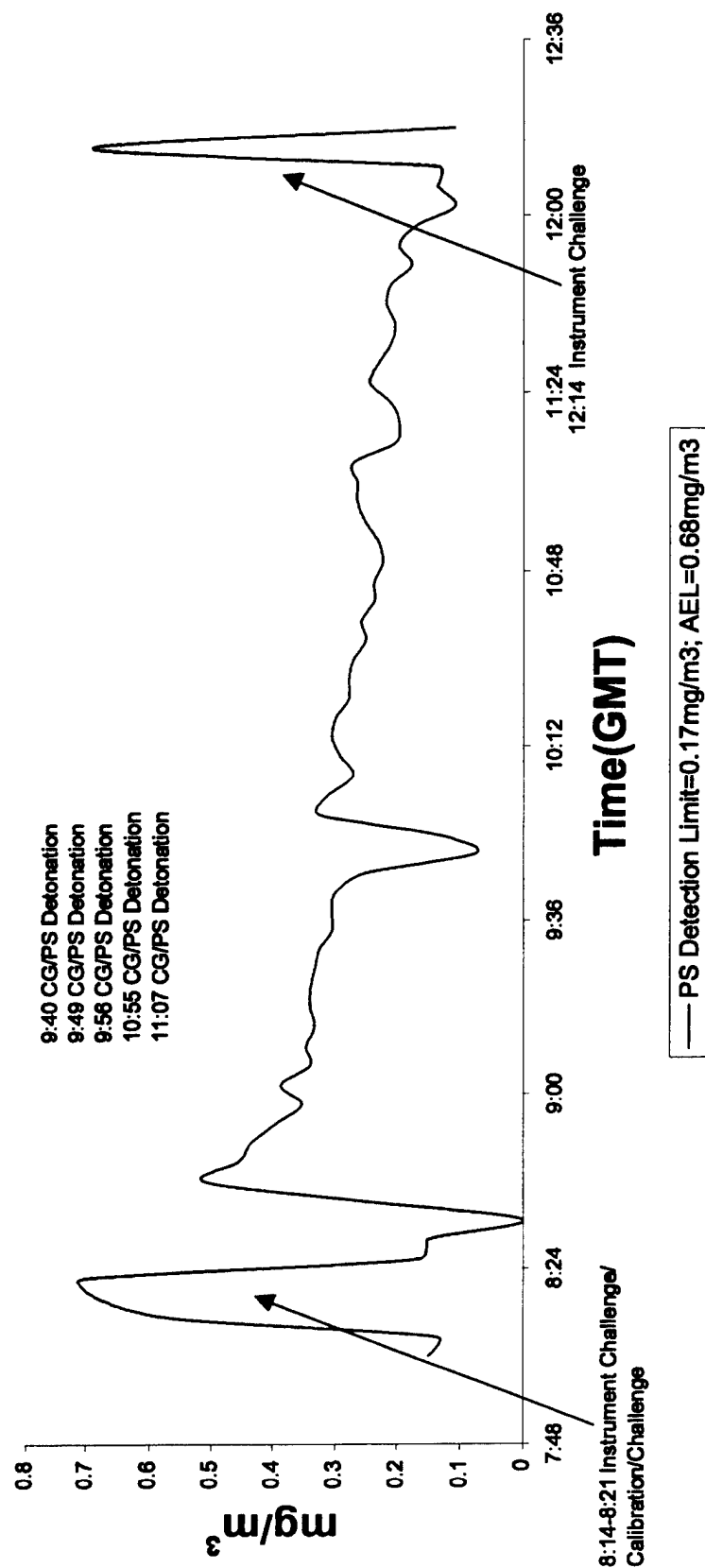


Figure 6.34

# July 3, 2001 MINICAMS Results for Mustard at the Exhaust Vent

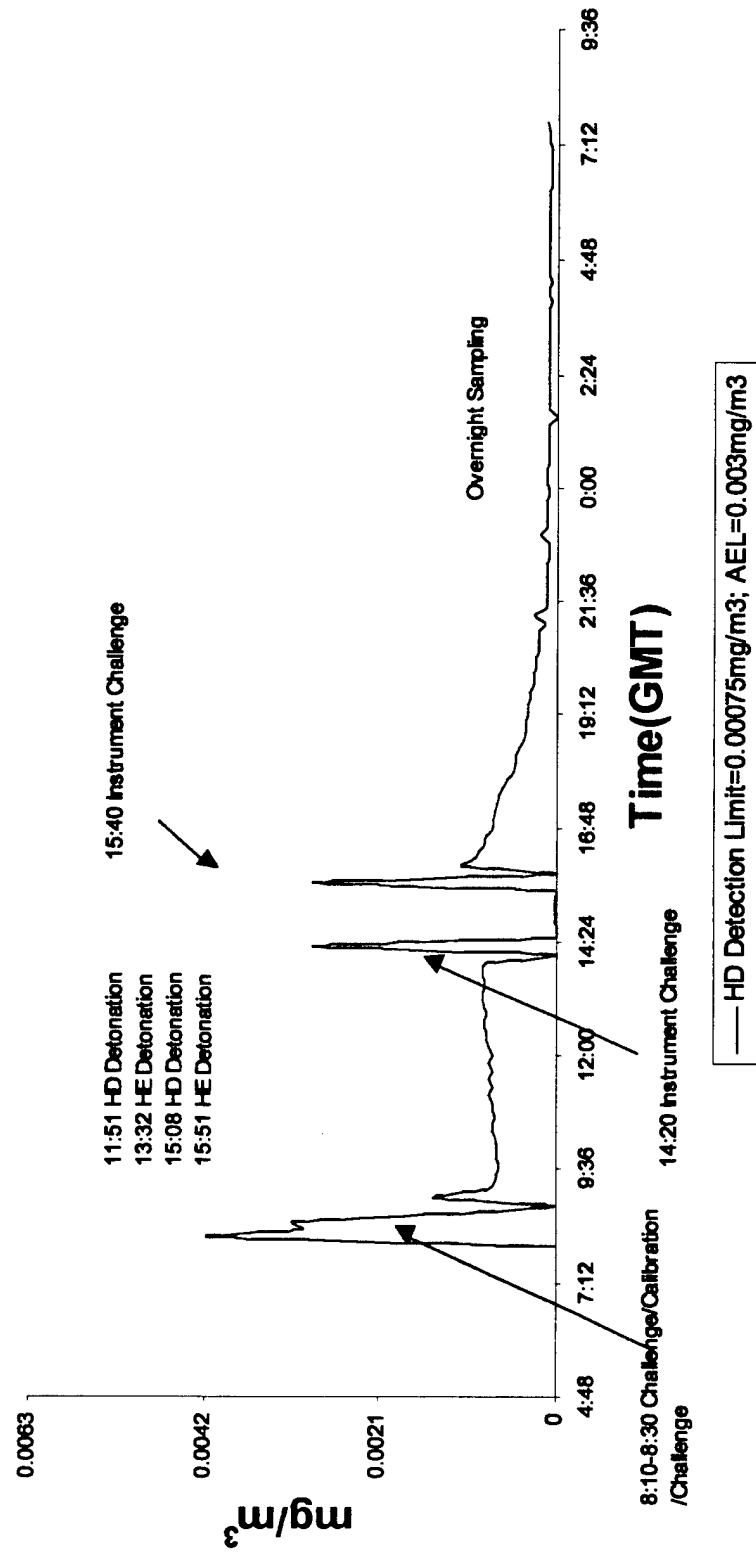


Figure 6.35

# July 3, 2001 MINICAMS Results for Mustard in the Expansion Chamber

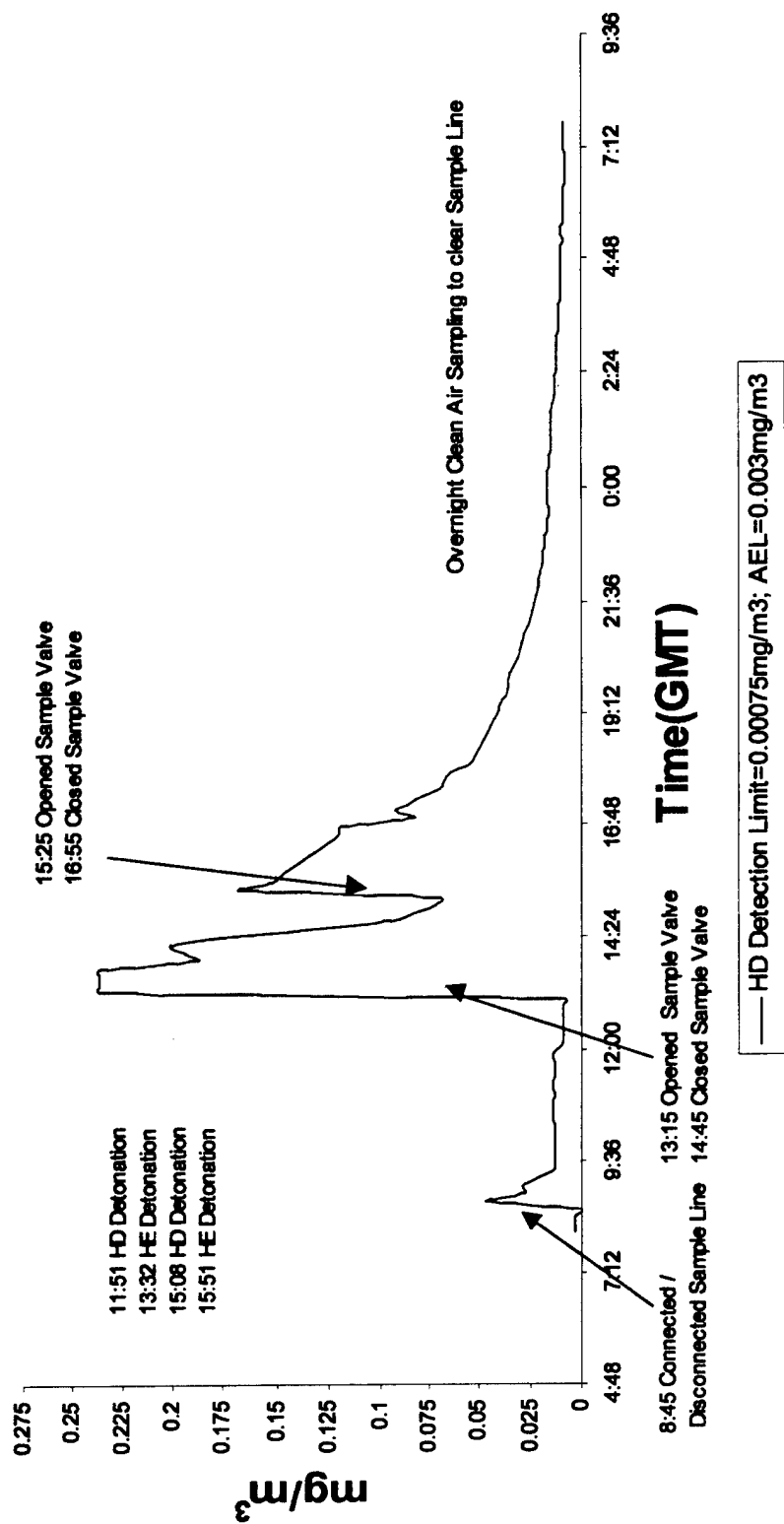


Figure 6.36

# July 3, 2001 MINICAMS Results for Mustard in the Monitoring Room

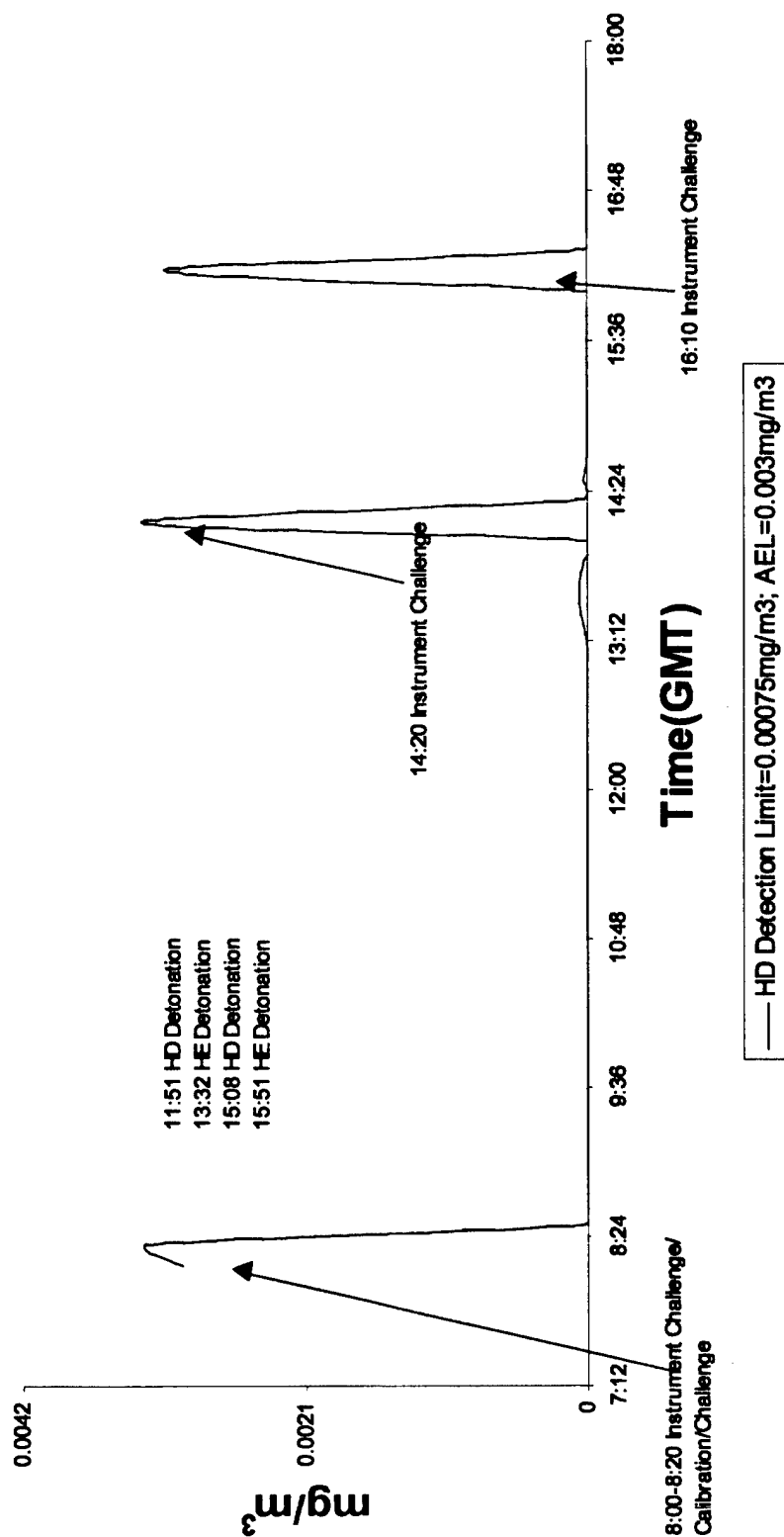


Figure 6.37

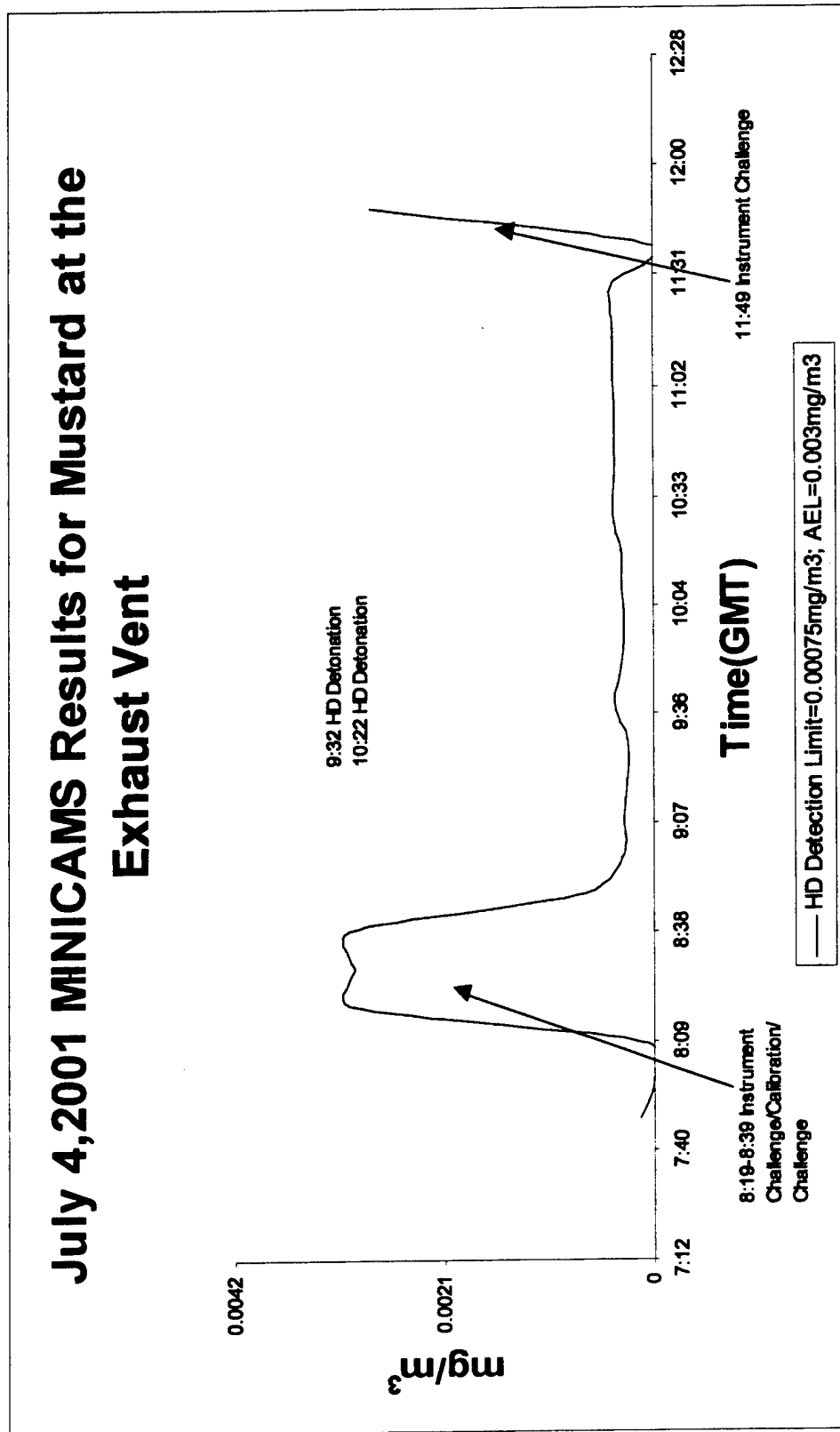


Figure 6.38

# July 4, 2001 MINICAMS Results for Mustard at the Expansion Chamber

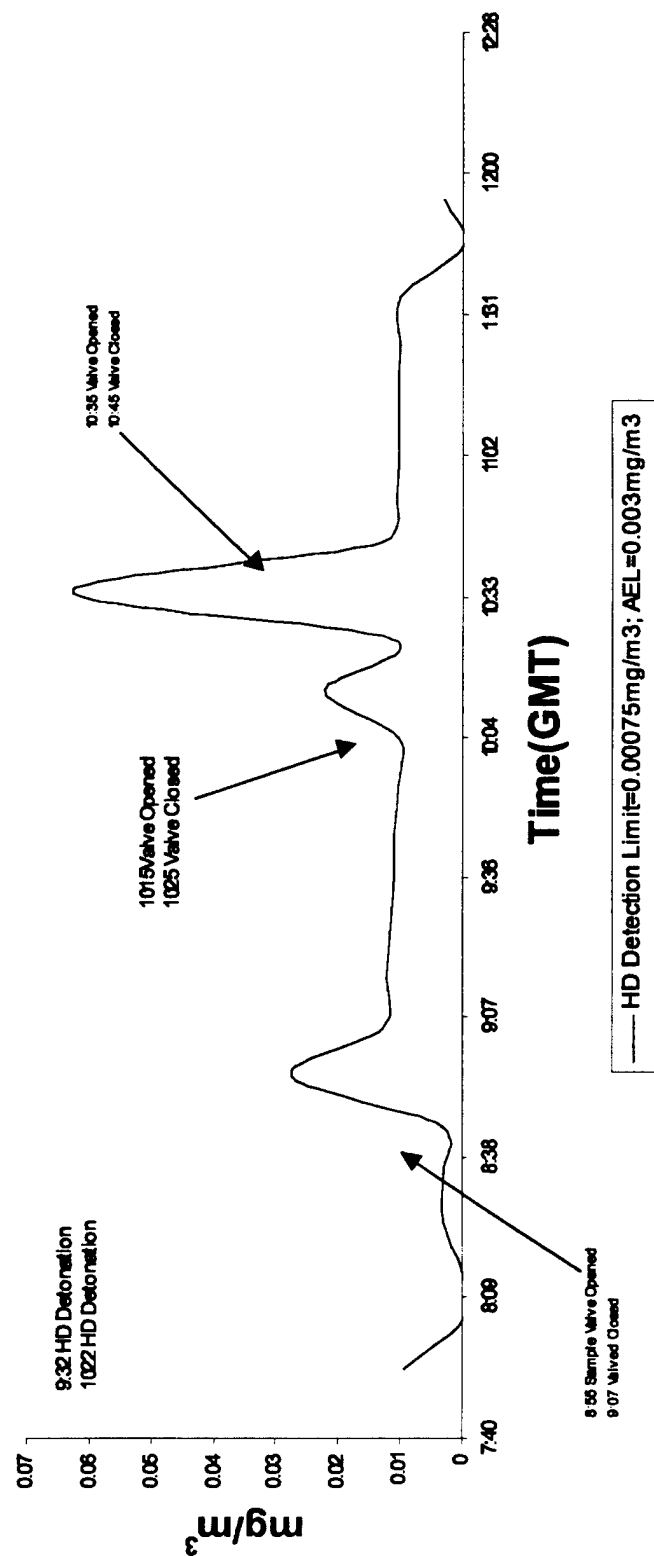


Figure 6.39

# July 4, 2001 MINICAMS Results for Mustard in the Monitoring Room

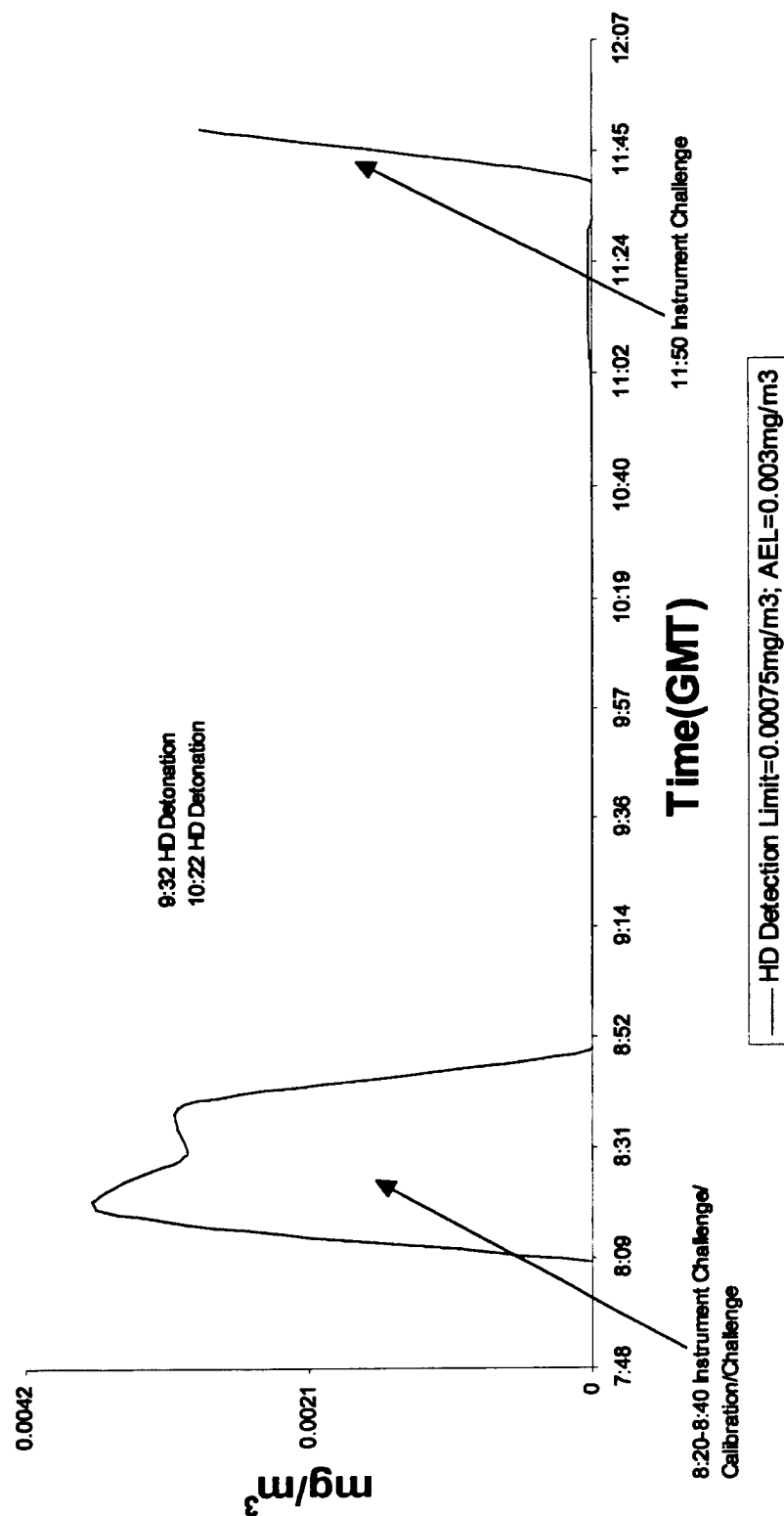


Figure 6.40

# July 5, 2001 MINICAMS Results for Phosgene at the Exhaust Vent

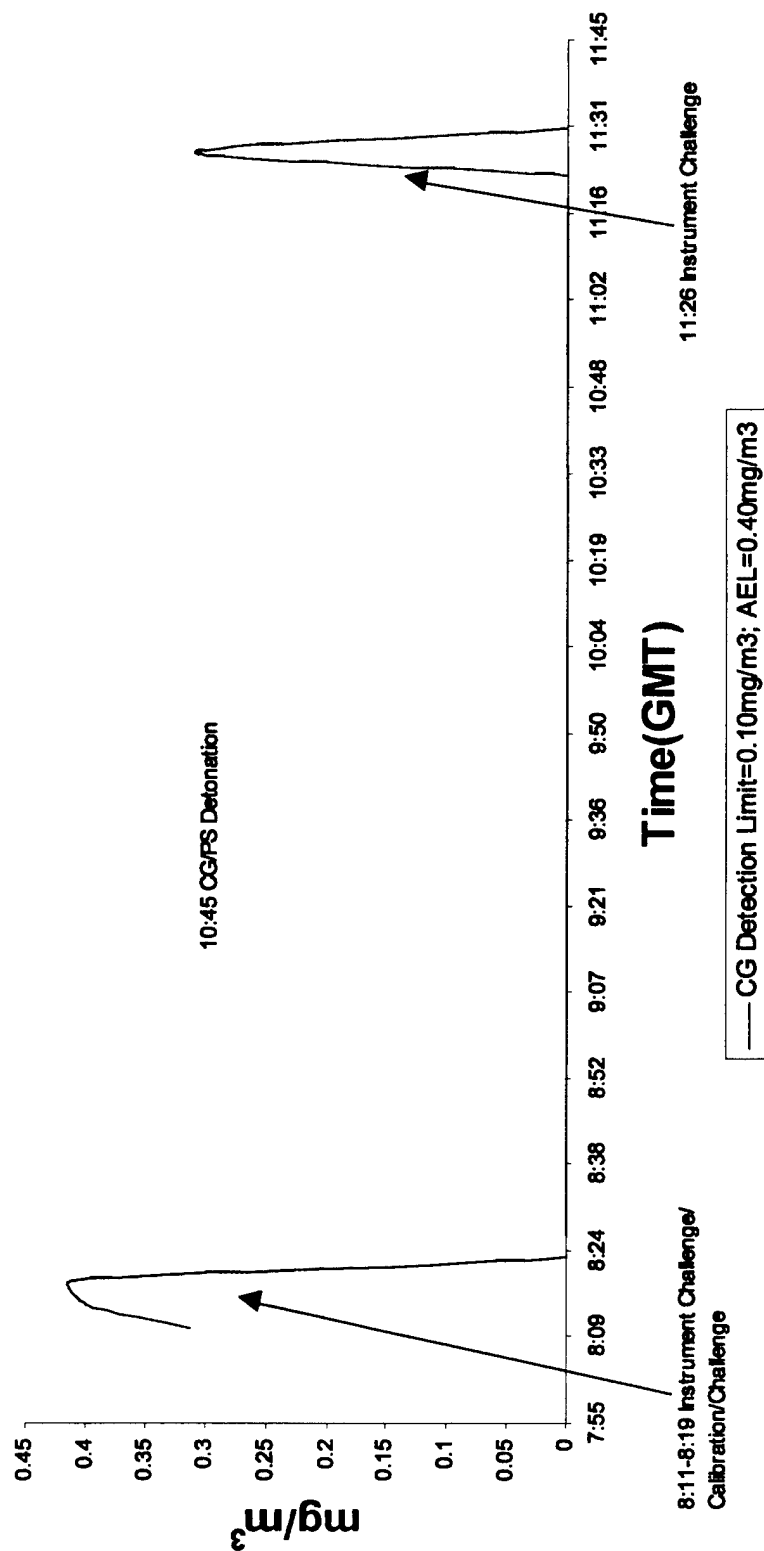




Figure 6.41

# July 5, 2001 MINICAMS Results for Chloropicrin at the Exhaust Vent

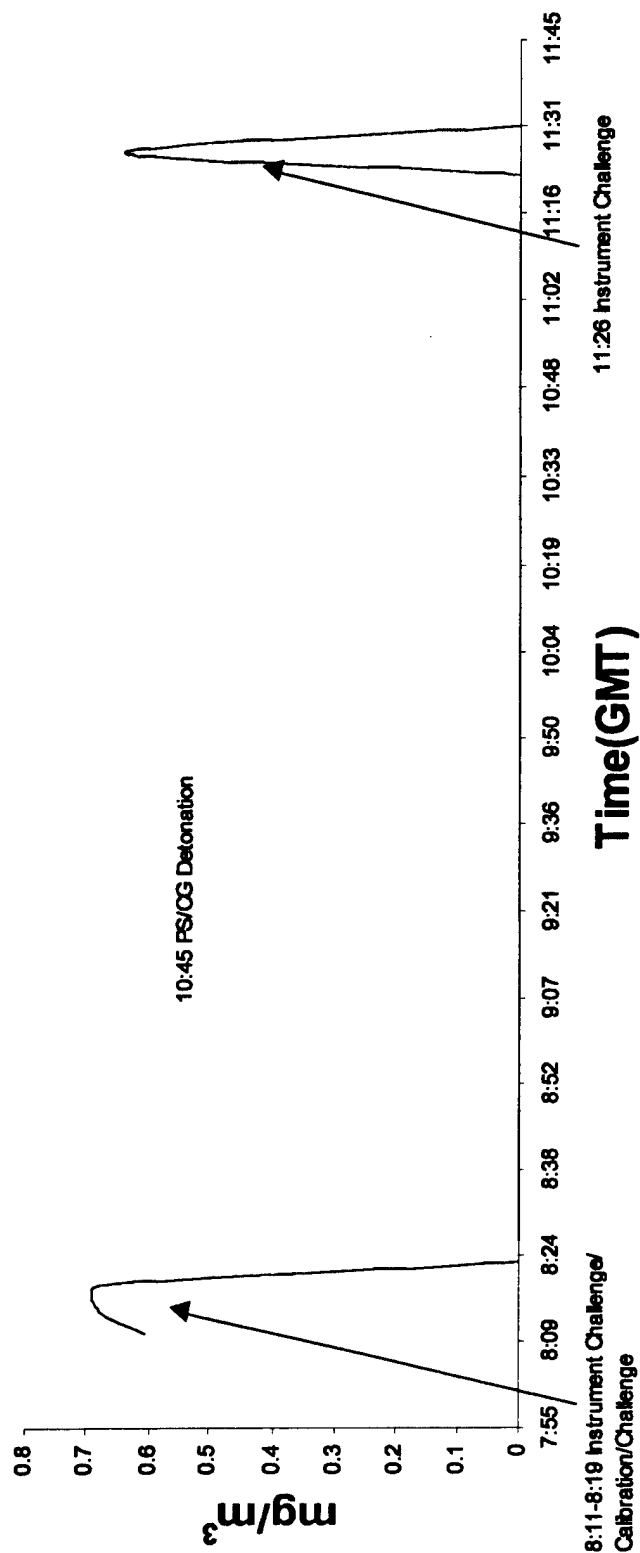


Figure 6.42

# July 5, 2001 MINICAMS Results for Phosgene at the Expansion Chamber

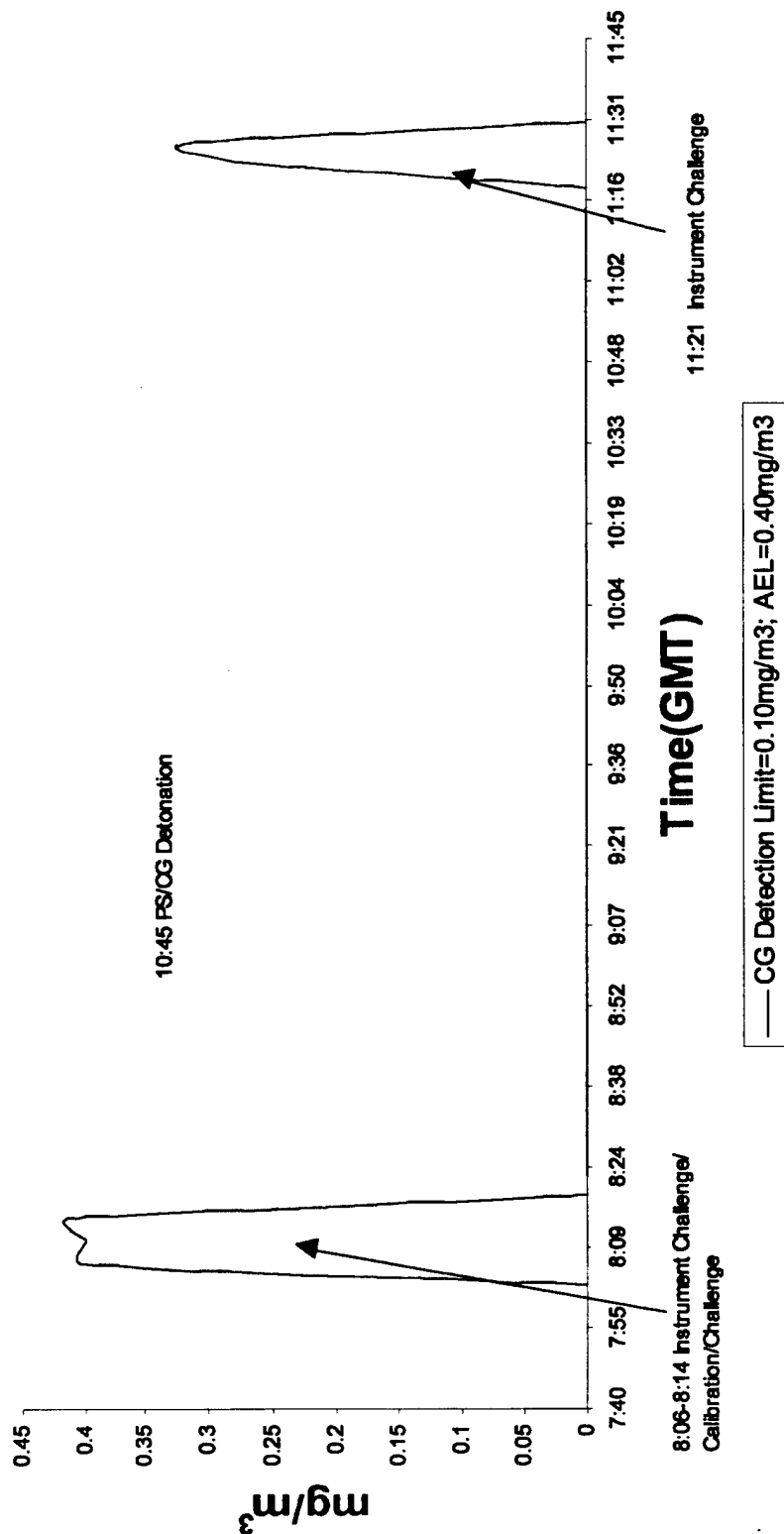
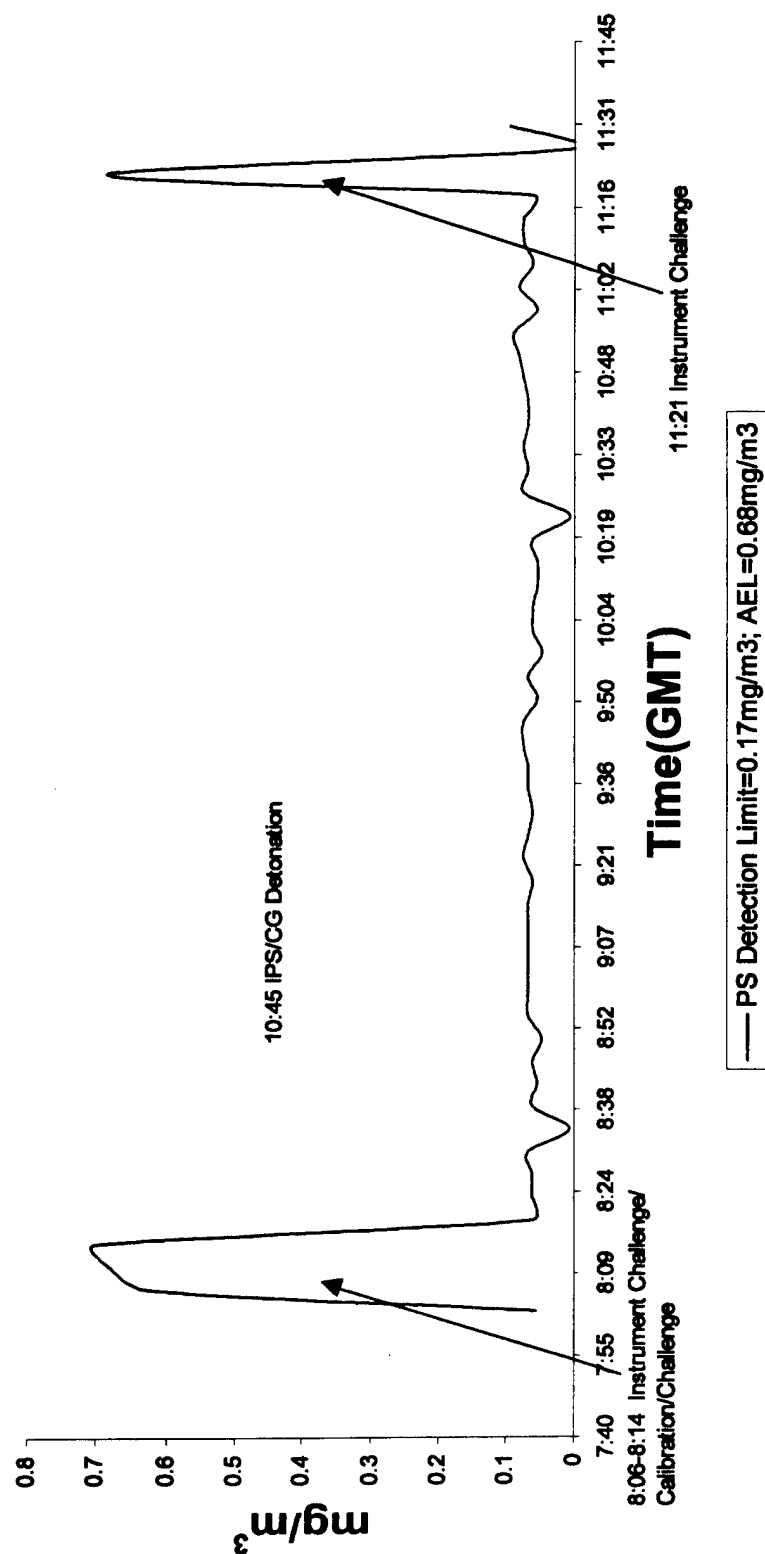


Figure 6.43

# July 5, 2001 MINICAMS Results for Chloropicrin at the Expansion Chamber



**APPENDIX 7.**  
**DAAMS RESULTS FOR HD**

$AEI = 0.003 \text{ mg/m}^3$   
 Detection Limit =  $0.000167 \text{ mg/m}^3$

Order of Detonation	Date	Type	Idf Number	North Perimeter	South Perimeter	East Perimeter	West Perimeter	Expansion Chamber Internal	Exhaust Duct	Exhaust Vent	Door Exhaust	DEMIL Personnel	MOD Personnel
1	14-May	N/A	N/A	ND	ND	ND	ND	ND	ND	N/A	ND	N/A	N/A
2		HE	N/A	ND	ND	ND	ND	ND	ND	N/A	ND	N/A	N/A
6	16-May	HD	2797	ND	ND	ND	ND	0.06883	ND	N/A	ND	N/A	N/A
7			2812	ND	ND	ND	ND	0.03142	ND	N/A	ND	N/A	N/A
8			2795	ND	ND	ND	ND	0.01333	ND	N/A	ND	N/A	N/A
9			N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
10	17-May	DA	3166	N/A	N/A	N/A	N/A	0.065	ND	N/A	ND	N/A	N/A
11			3167	N/A	N/A	N/A	N/A	0.01258	ND	N/A	ND	N/A	N/A
12			3168	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
13	18-May	CG	3102	N/A	N/A	N/A	N/A	0.001	ND	N/A	N/A	N/A	N/A
14			3117	N/A	N/A	N/A	N/A	0.00061	ND	N/A	N/A	N/A	N/A
15	21-May	DA	3151	N/A	N/A	N/A	N/A	0.0003	ND	N/A	N/A	N/A	N/A
16		HD	2717	ND	ND	ND	ND	0.0045	ND	N/A	ND	N/A	N/A
29-39	15-June	DA	3172; 3221; 3020; 3212; 3223; 3205; 3201; 3206; 3208; 3243	NA	NA	NA	NA	N/A	N/A	N/A	ND	N/A	N/A
40-43			3242; 3293; 3237; 3235	NA	NA	NA	NA	N/A	N/A	N/A	ND	N/A	N/A

Order of Detonation	Date	Type	Idf Number	North Perimeter	South Perimeter	East Perimeter	West Perimeter	Expansion Chamber Internal	Exhaust Duct	Exhaust Vent	Door Exhaust	DEMIL Personnel	MOD Personnel
44	20-June	HD	2665	ND	ND	ND	ND	OVER-LOADED	ND	ND	0.0267	.0000825	0.06467
45			2788					OVER-LOADED					
46			2659	ND	ND	ND	ND	OVER-LOADED	OVER-LOADED	ND	0.0229		
47			2662					OVER-LOADED					
48	21-June		2780	ND	ND	ND	ND	OVER-LOADED	5.38	ND	OVER-LOADED	0.0146	0.52532
49			2807					OVER-LOADED					
50			3103					OVER-LOADED					
51			3104					OVER-LOADED					
52	22-June		3106	ND	ND	ND	ND	OVER-LOADED	N/A	ND	N/A	0.007288	0.30775
53	22-June	HD	3135					OVER-LOADED	N/A		N/A		
54		N/A	N/A					N/A	N/A		N/A		
55	25-June	N/A	N/A	ND	ND	ND	ND	N/A	N/A	ND	ND	0.082466	0.00015
72	3-July	HD	3043	ND	ND	ND	ND	0.000351	OVER-LOADED	ND	0.00063	N/A	N/A
73		HE	N/A					N/A	N/A		.00007	N/A	N/A
74		HD	3033	ND	ND	ND	ND	N/A	N/A	ND		N/A	N/A
75		HE	N/A					N/A	N/A			N/A	N/A
76	4-July	HD	3132	ND	ND	ND	ND	0.005454	0.0012	ND	OVER-LOADED	N/A	N/A
77			3056									N/A	N/A

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**APPENDIX 8.**  
**OPFTIR RESULTS**



Figure 8.1

## May 14, 2001 OPFTIR Monitoring for Carbon Monoxide over Detonation Chamber Door

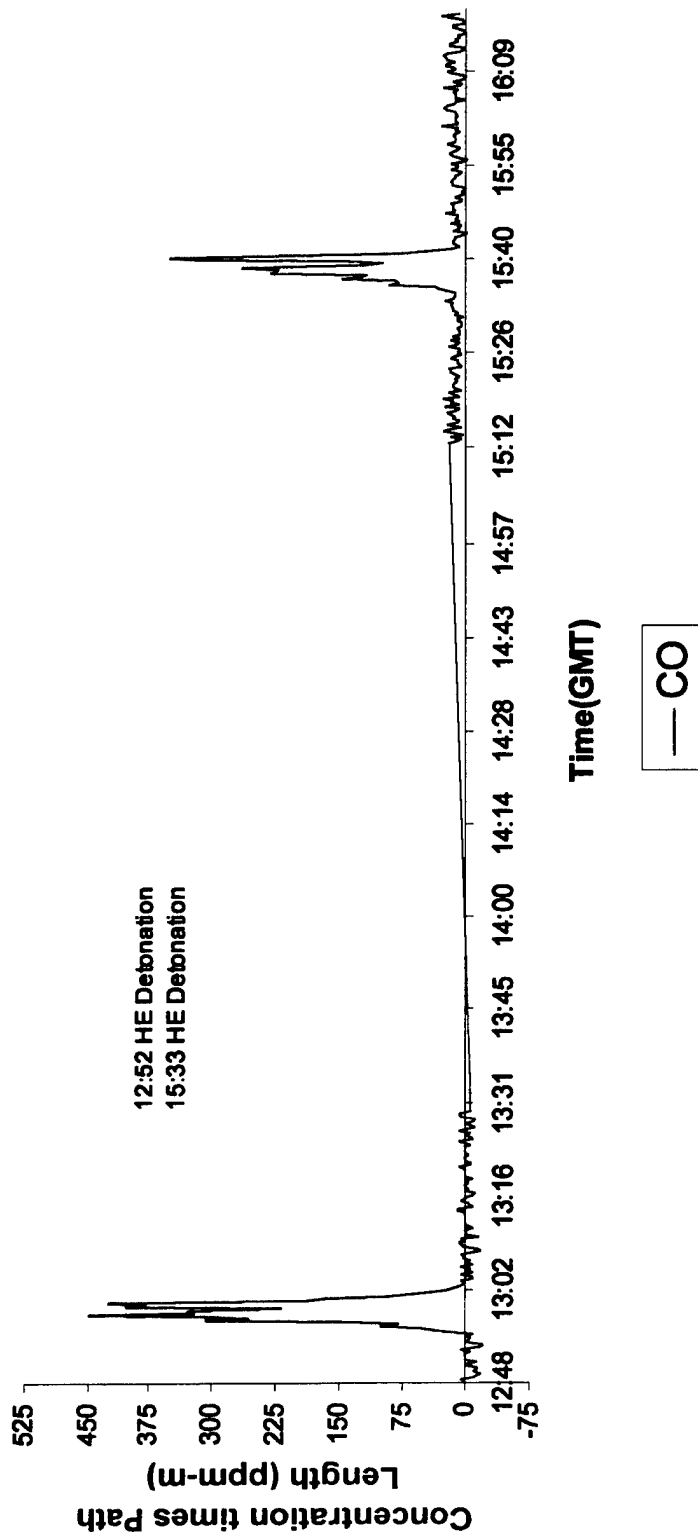


Figure 8.2

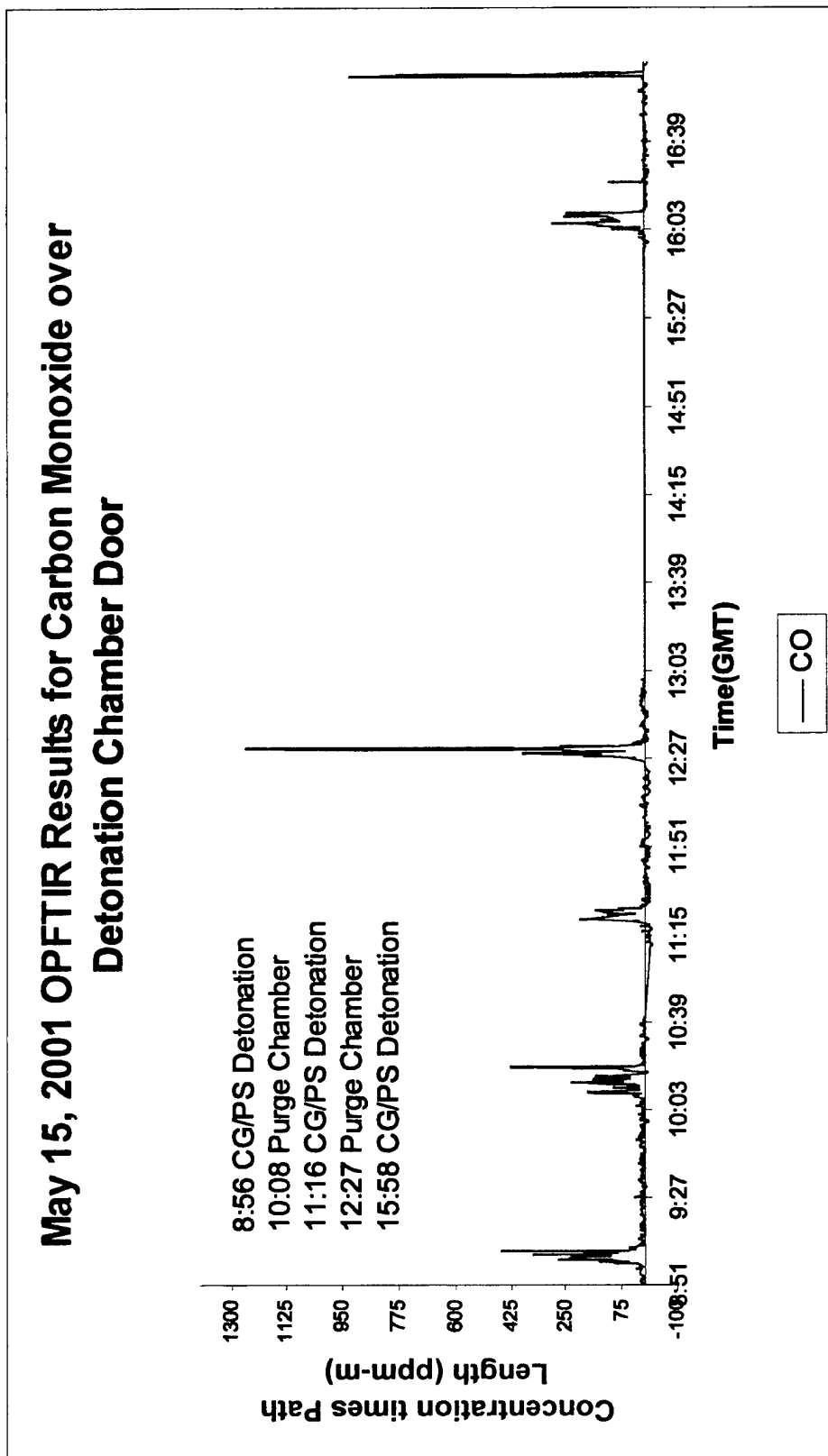


Figure 8.3

# **May 15, 2001 OPFTIR Results for Ethylene and Methane over Detonation Chamber Door**

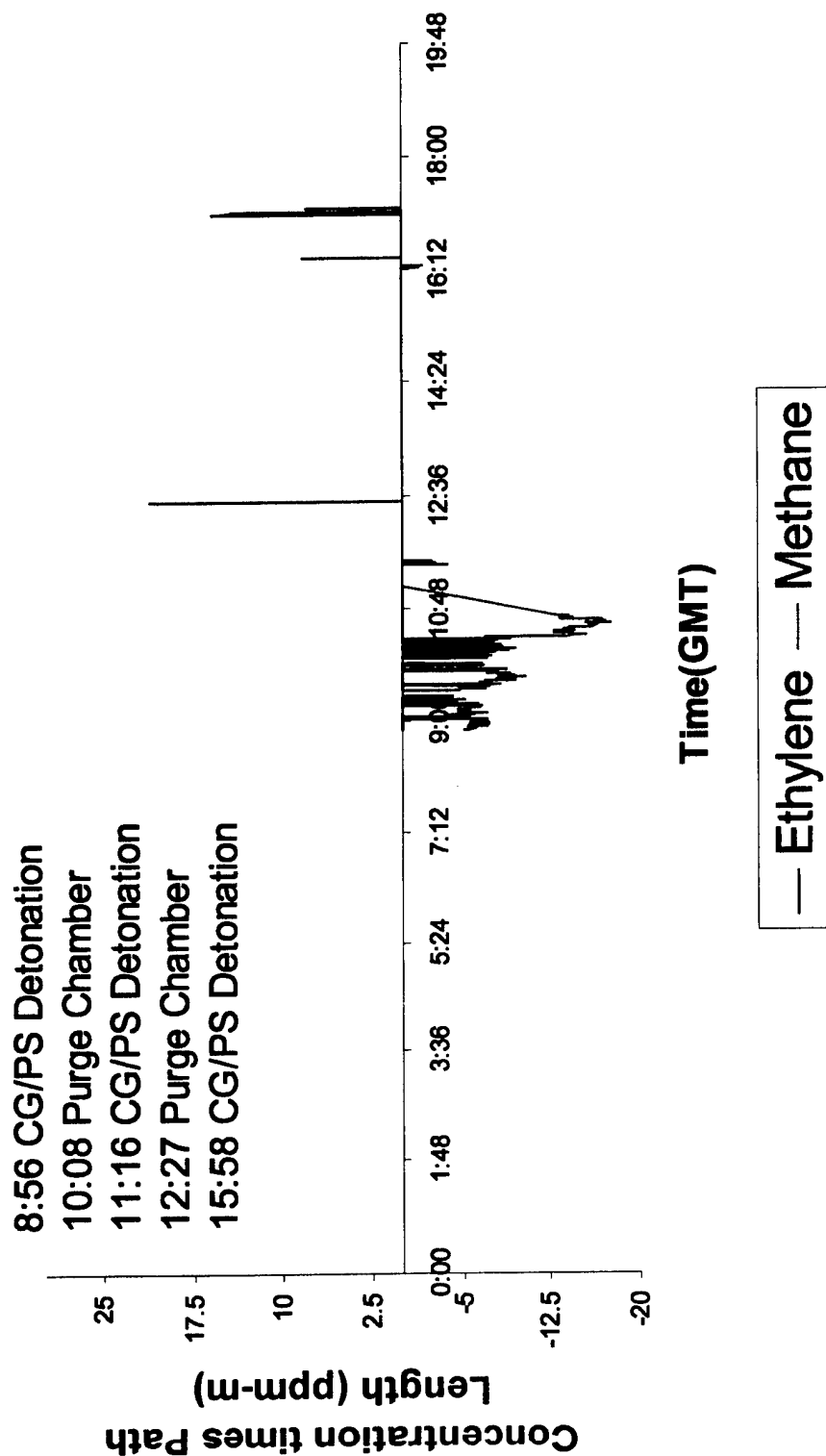


Figure 8.4

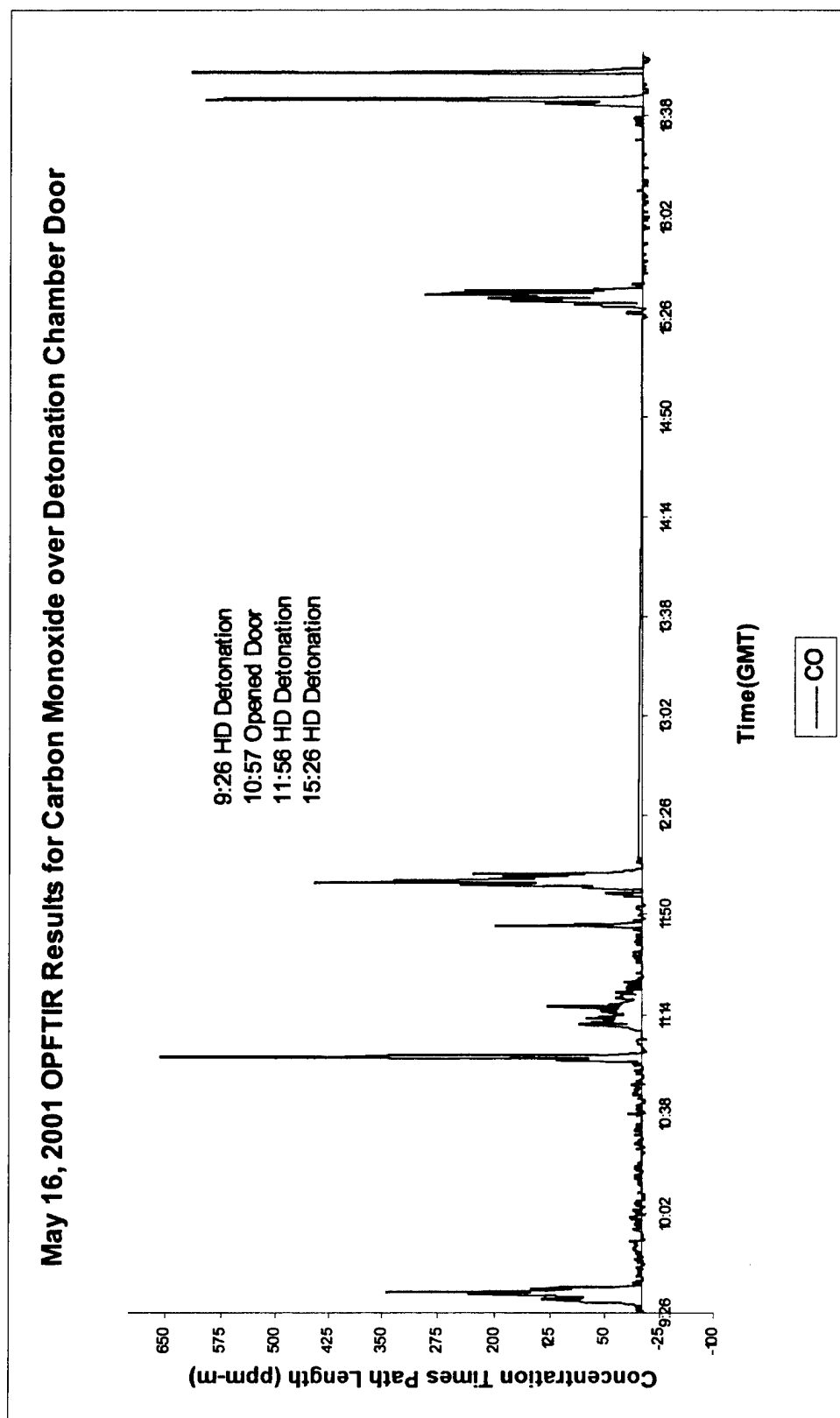


Figure 8.5

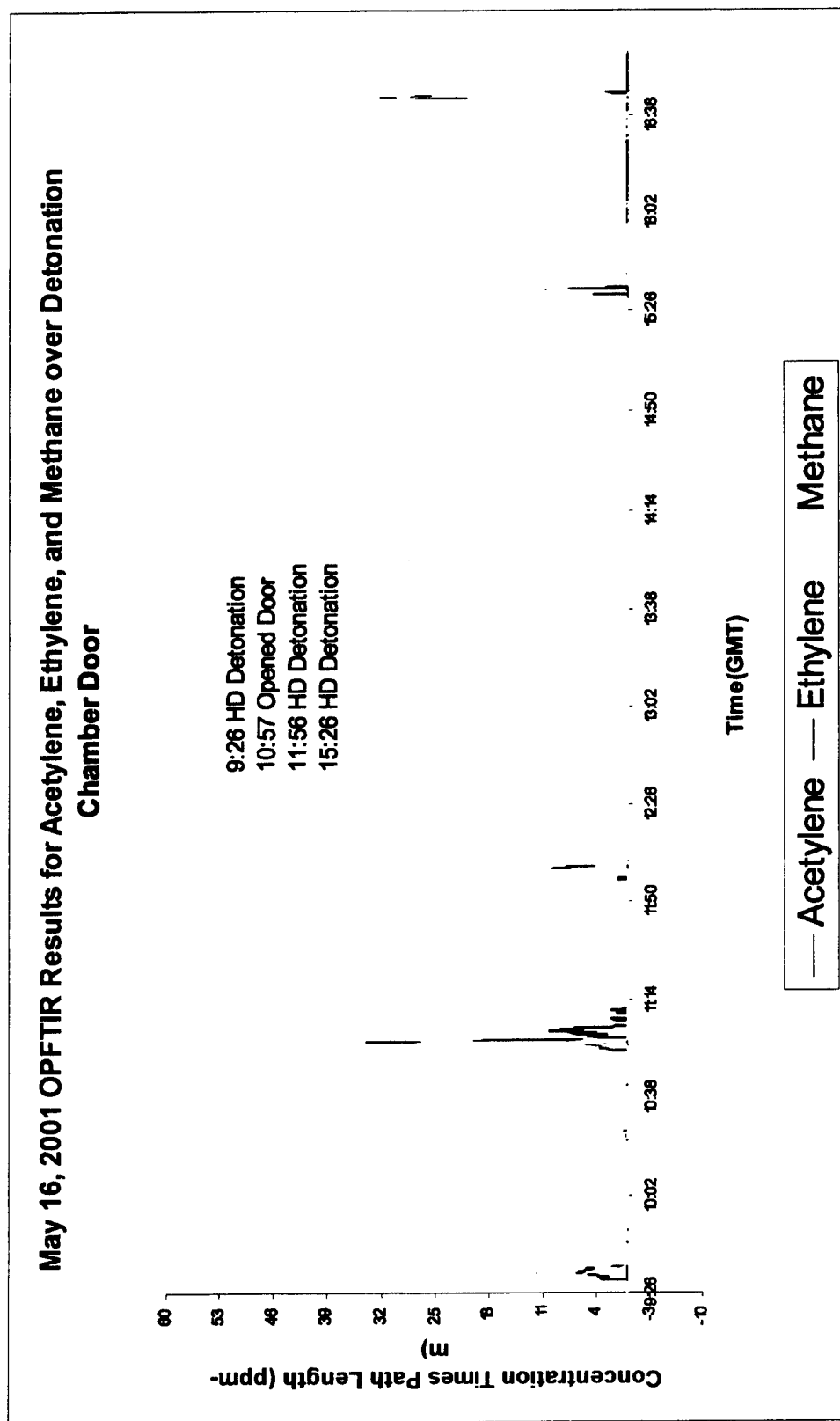


Figure 8.6

# May 17, 2001 OPFTIR Results for Carbon Monoxide over Detonation Chamber Door

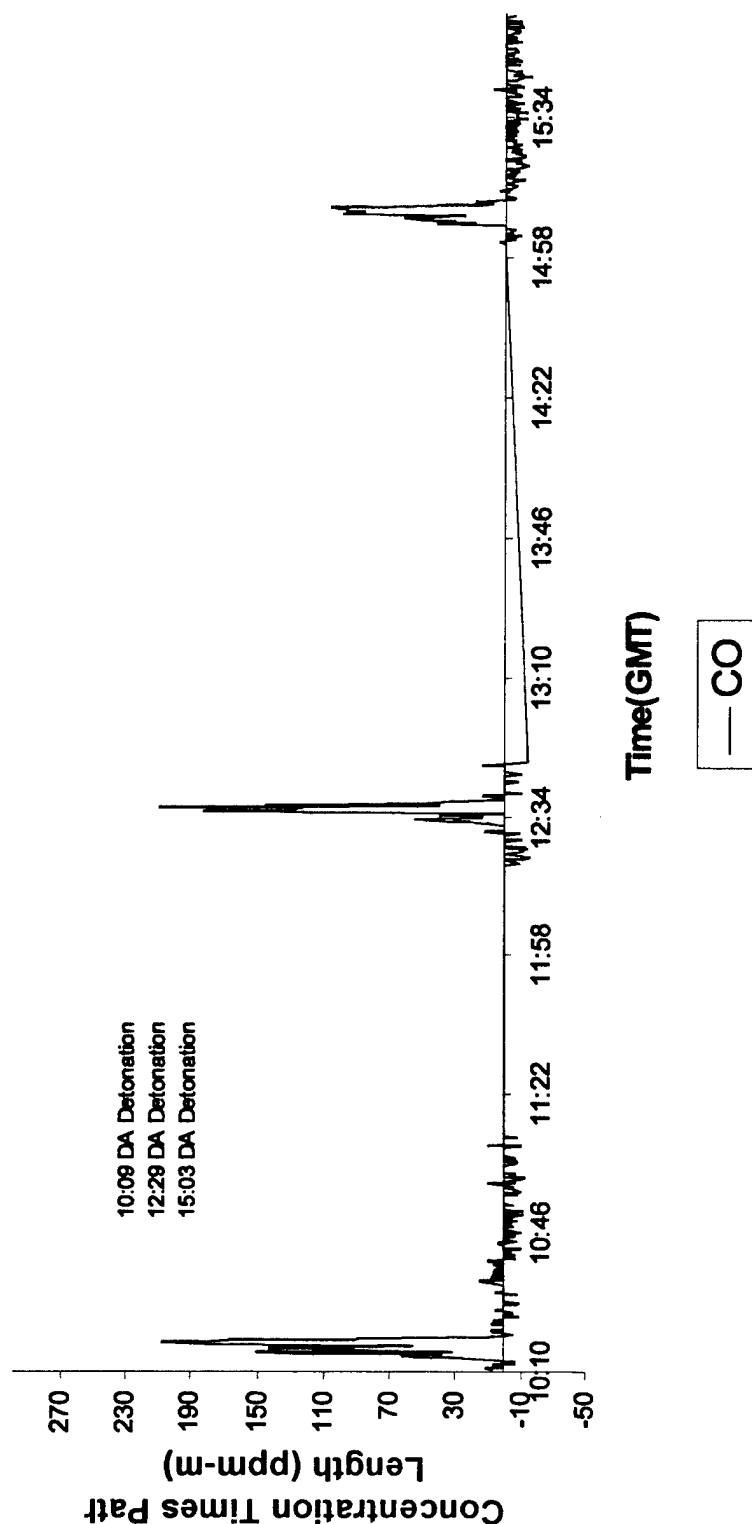


Figure 8.7

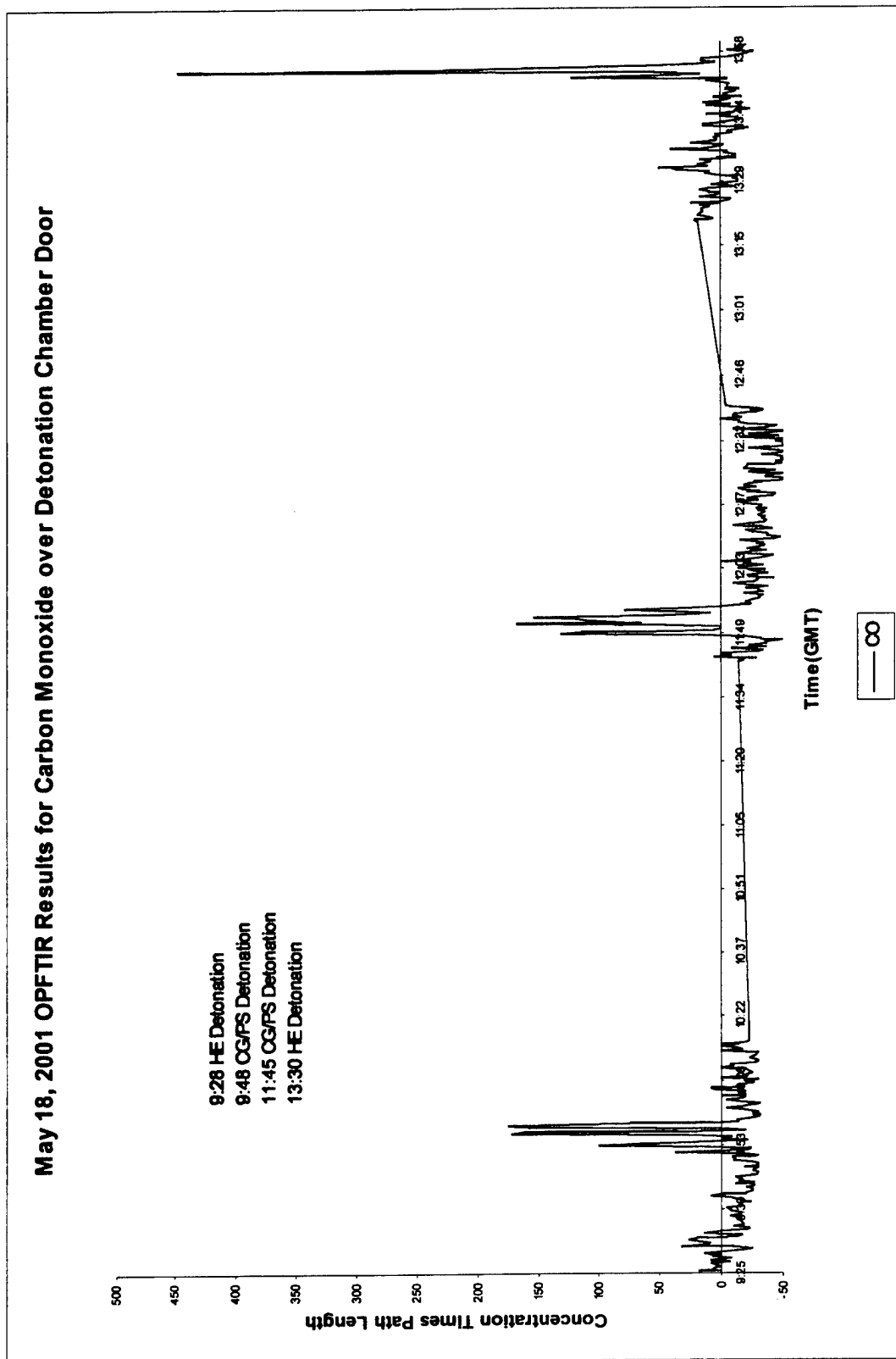


Figure 8.8

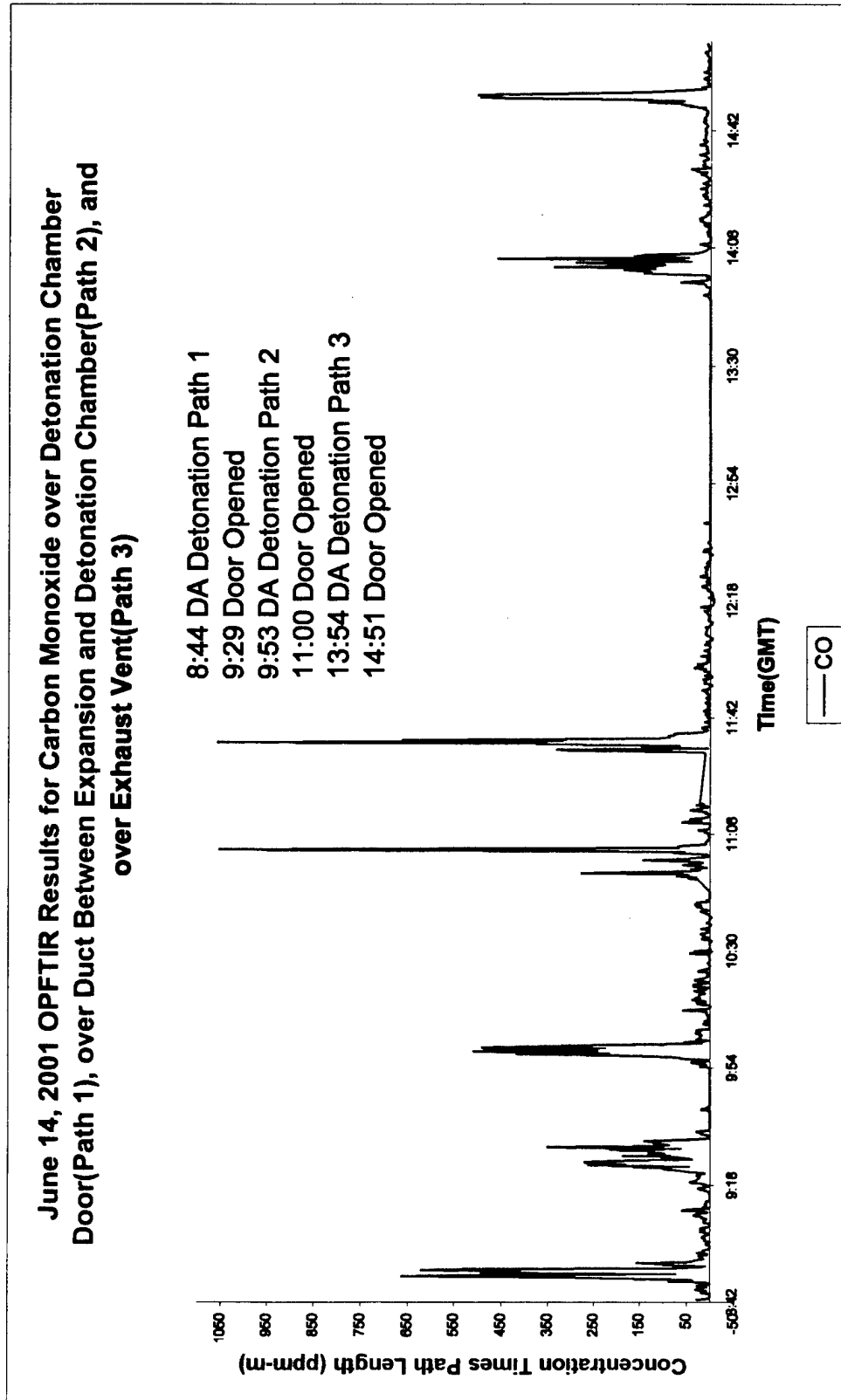




Figure 8.9

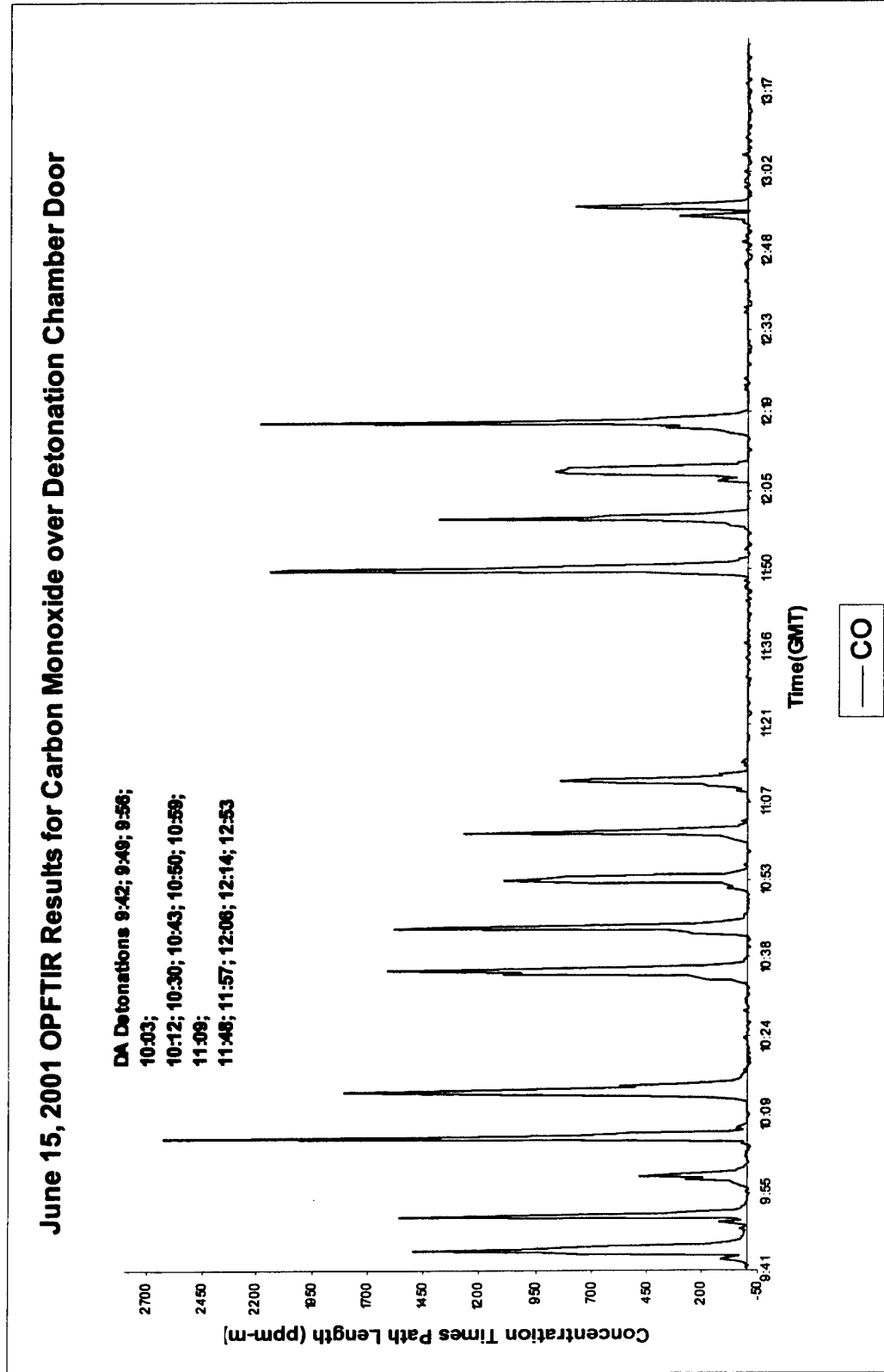


Figure 8.10

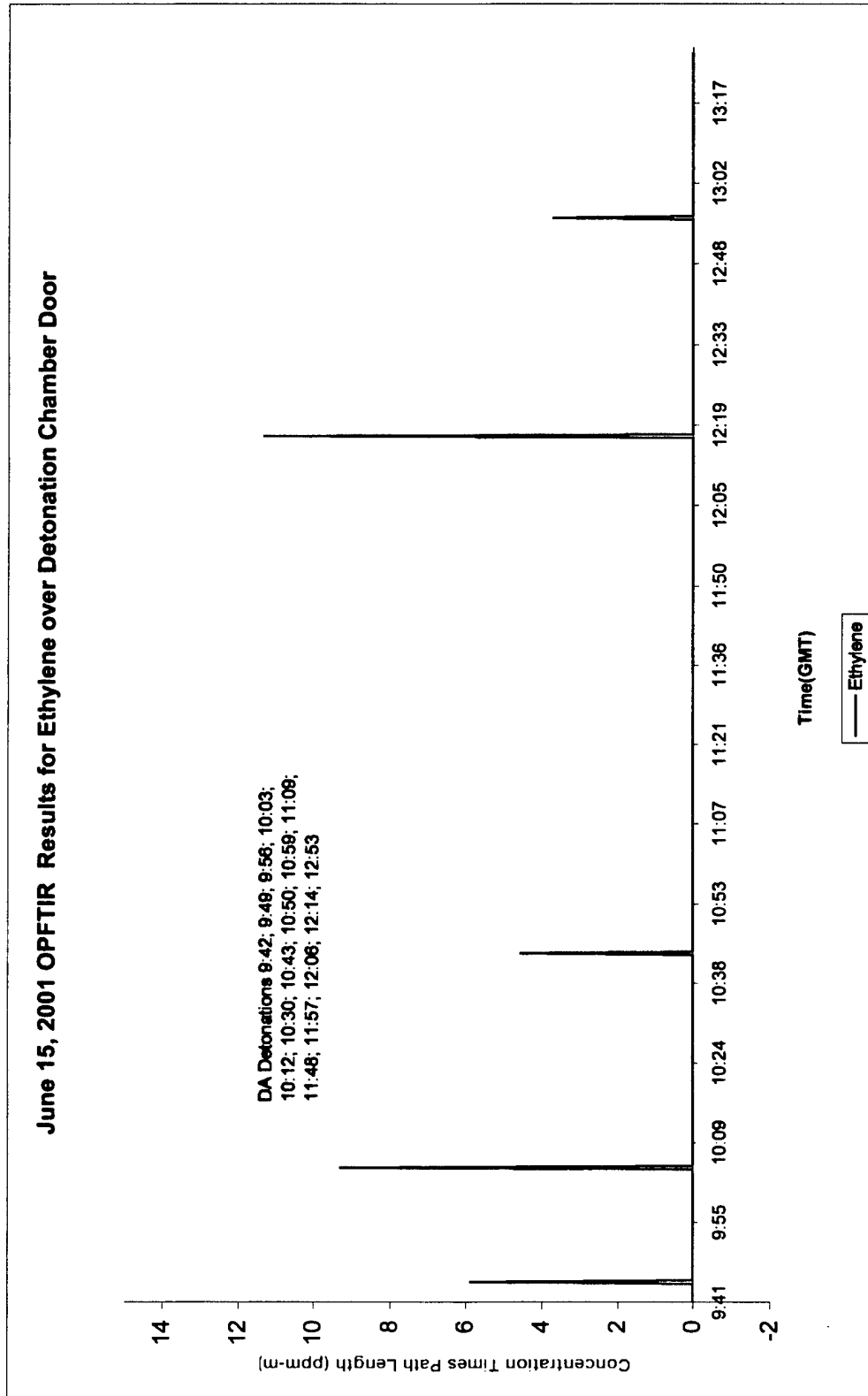


Figure 8.11

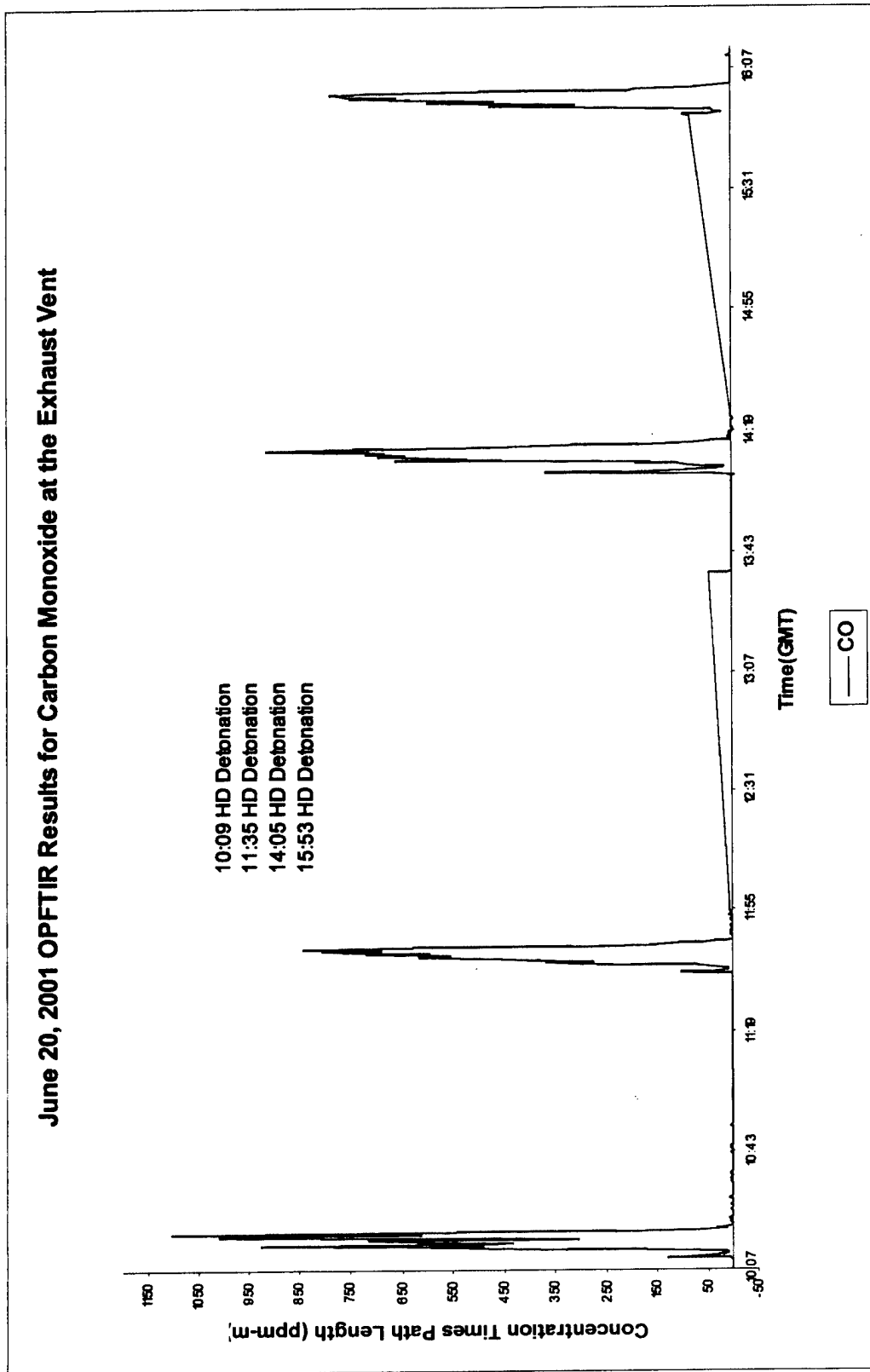


Figure 8.12

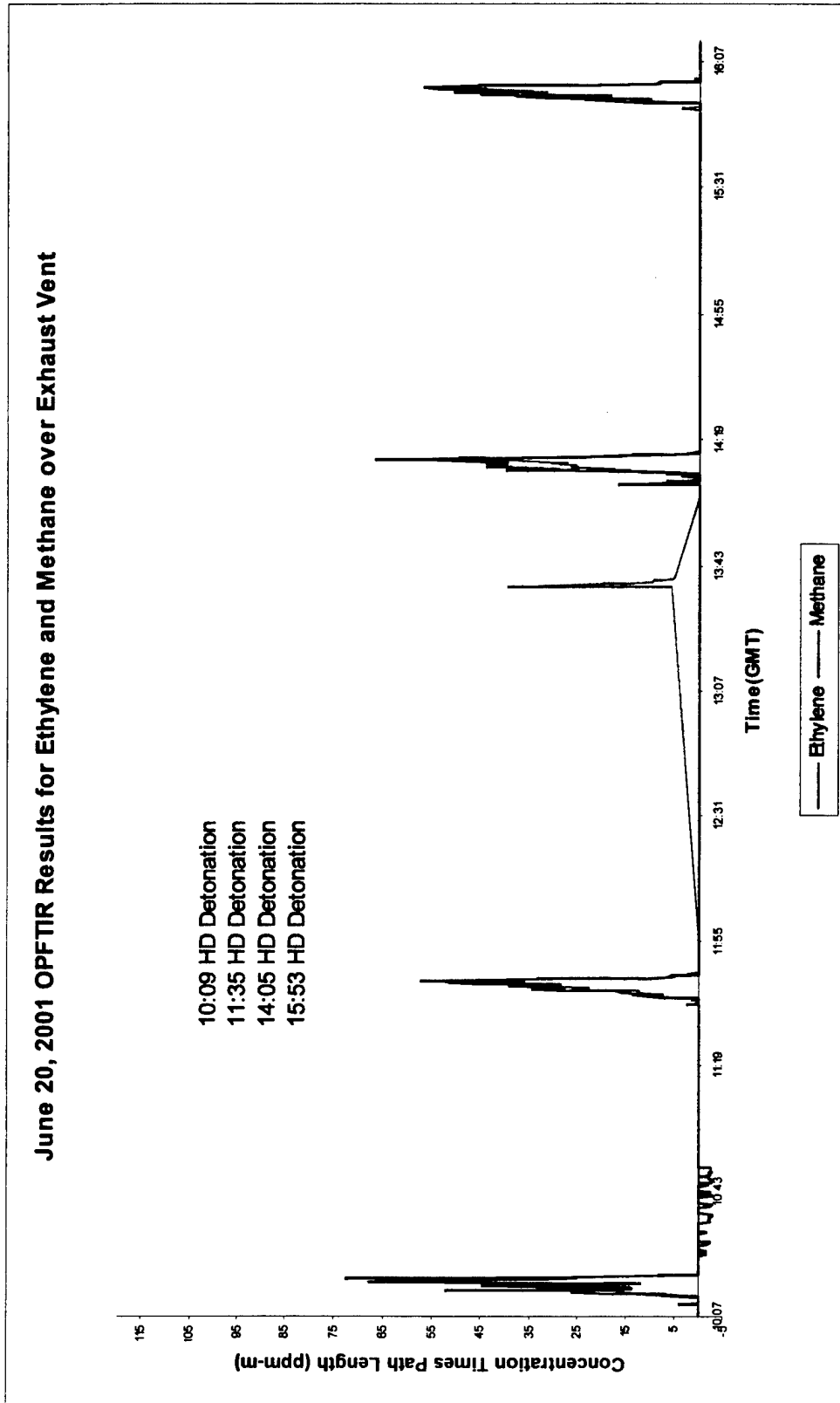


Figure 8.13

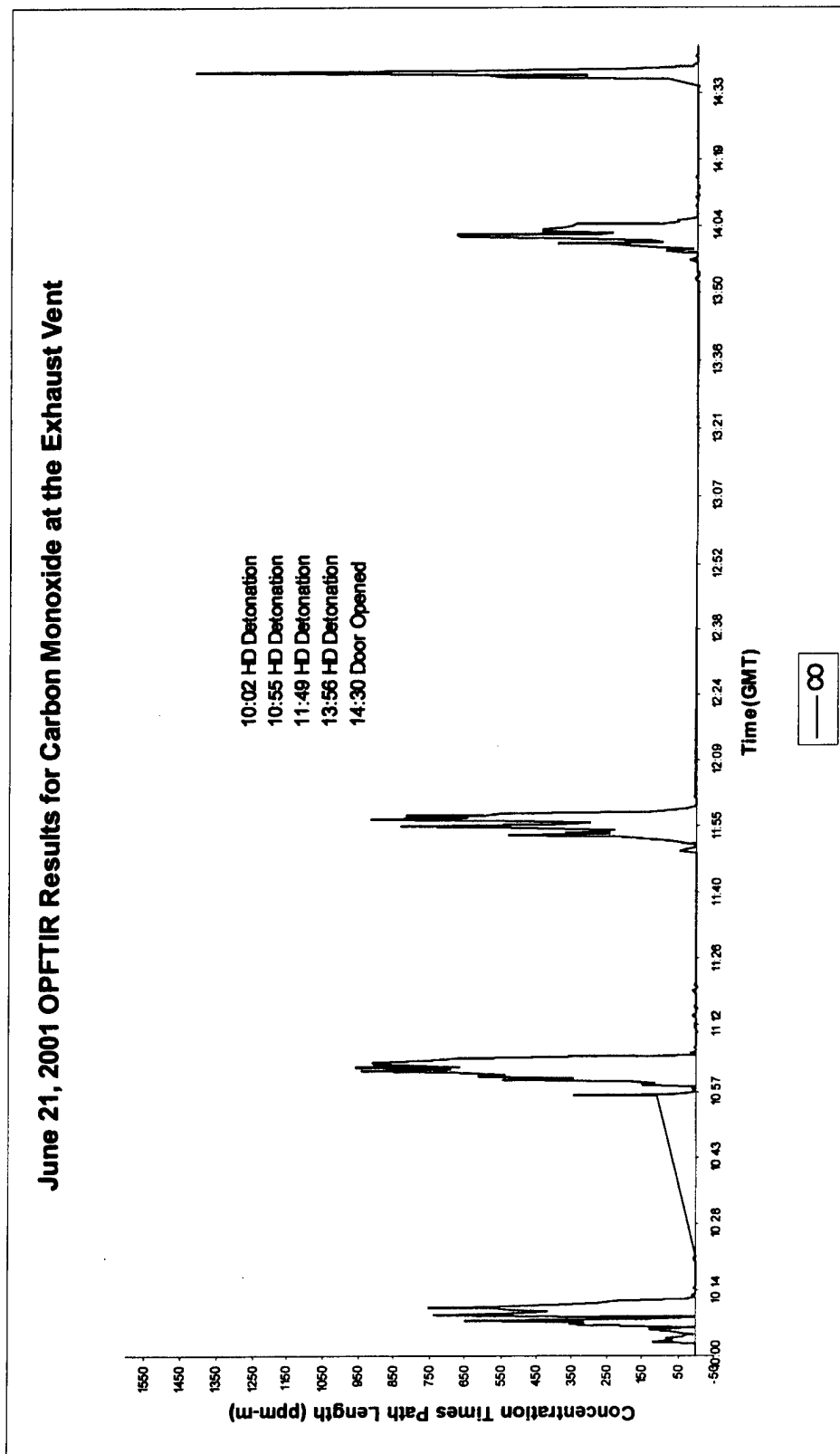


Figure 8.14

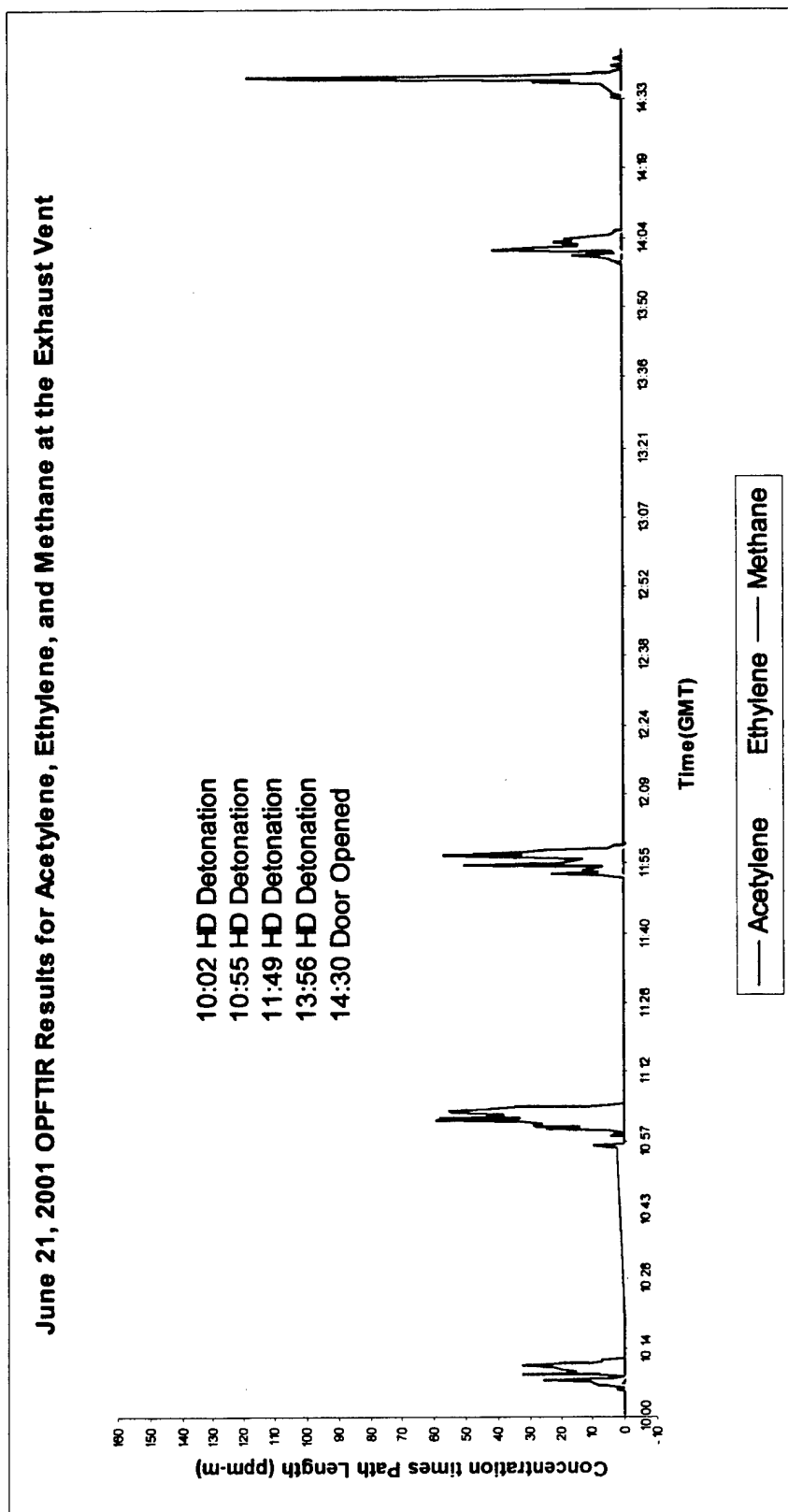


Figure 8.15

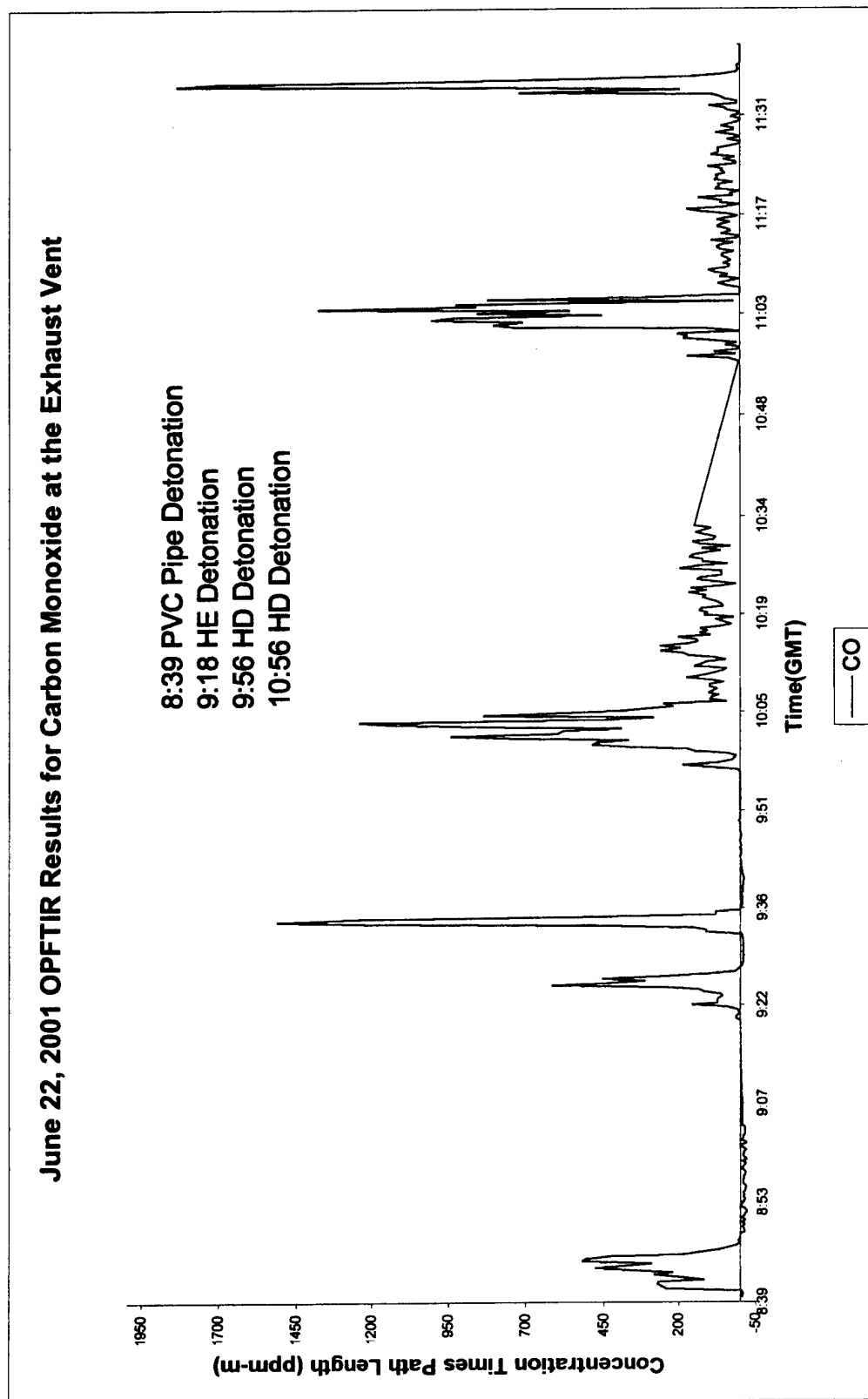
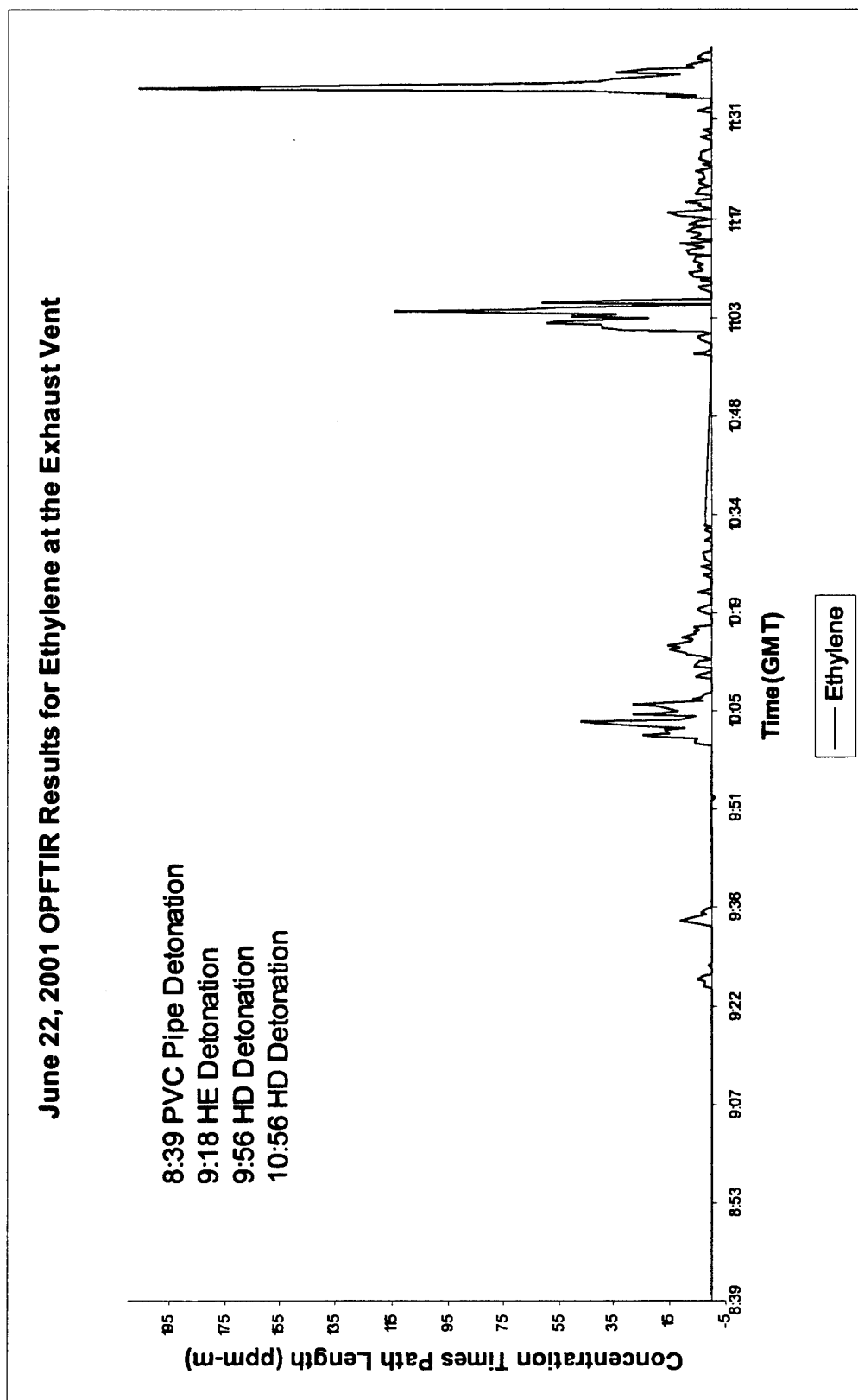


Figure 8.16





**Figure 8.17**

**June 27, 2001 OPFTIR Results for Carbon Monoxide at the Exhaust Vent**

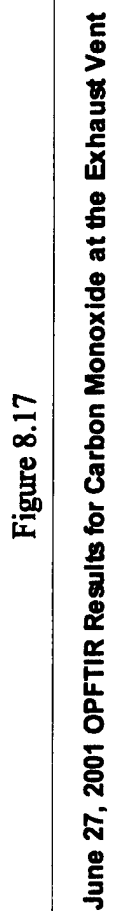


Figure 8.18

# June 27, 2001 OPFTIR Results for Acetylene, Ethylene, and Methane at the Exhaust Vent

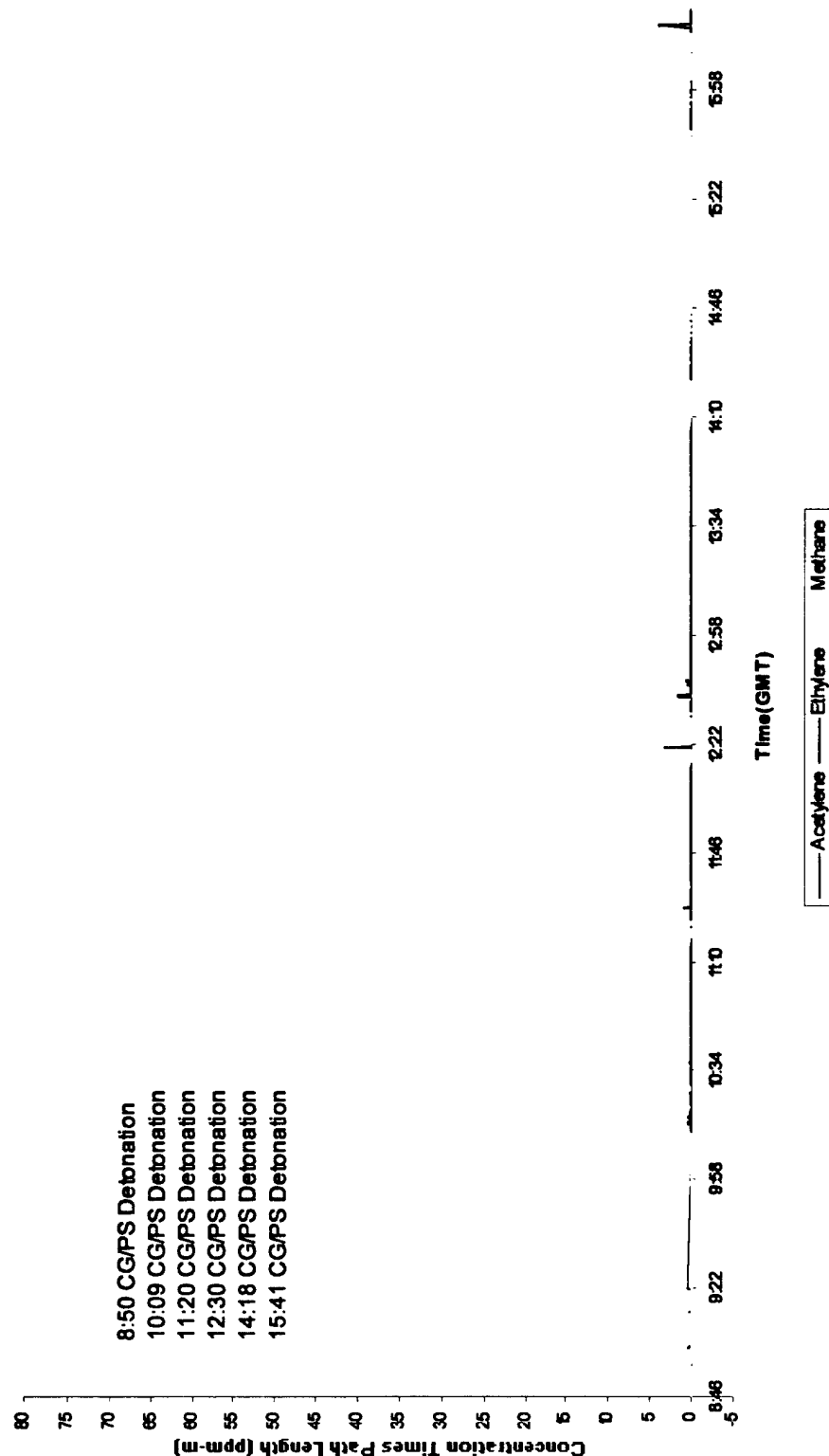


Figure 8.19

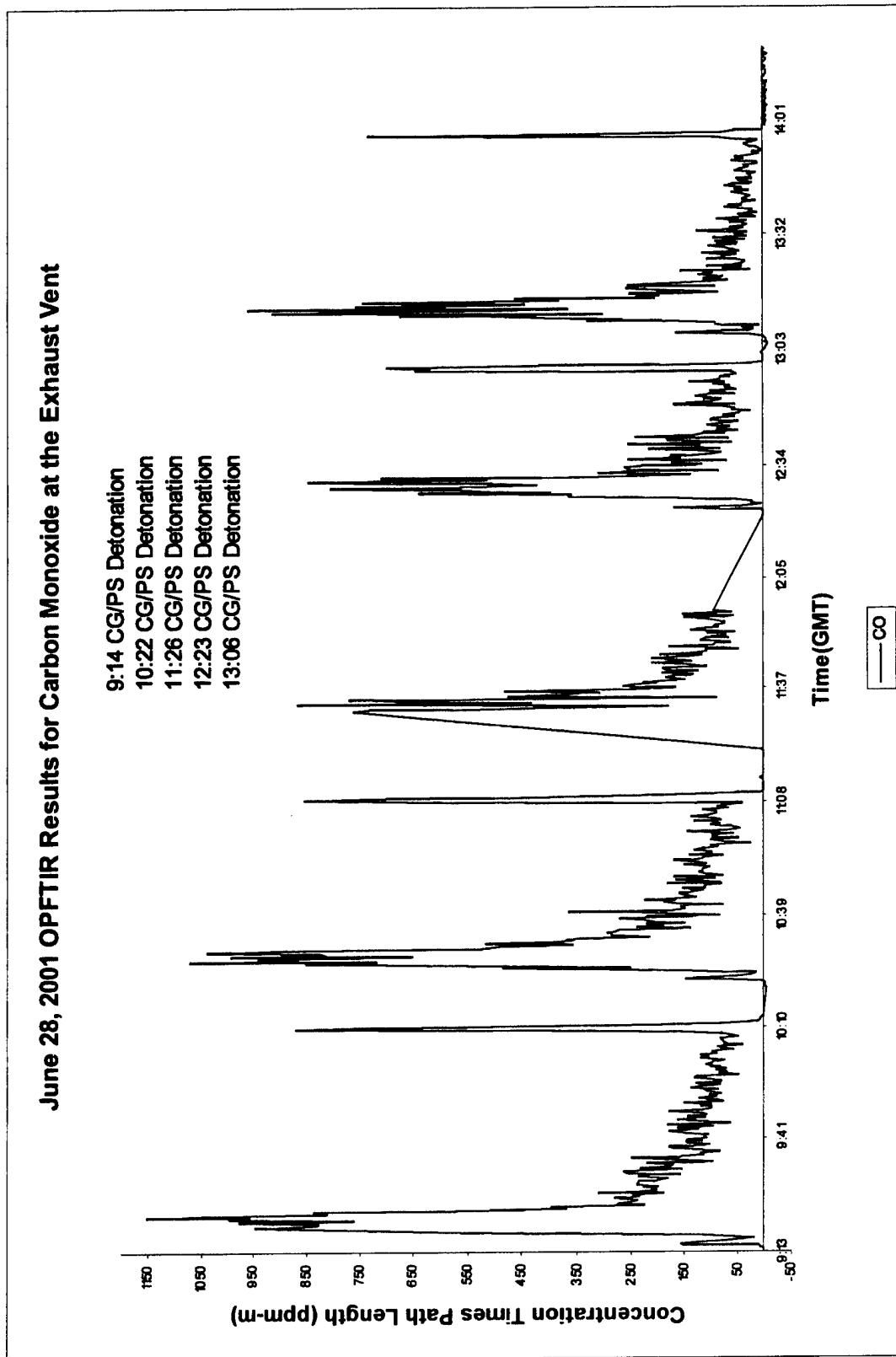


Figure 8.20

June 28, 2001 OPFTIR Results for Ethylene and Methane at the Exhaust Vent

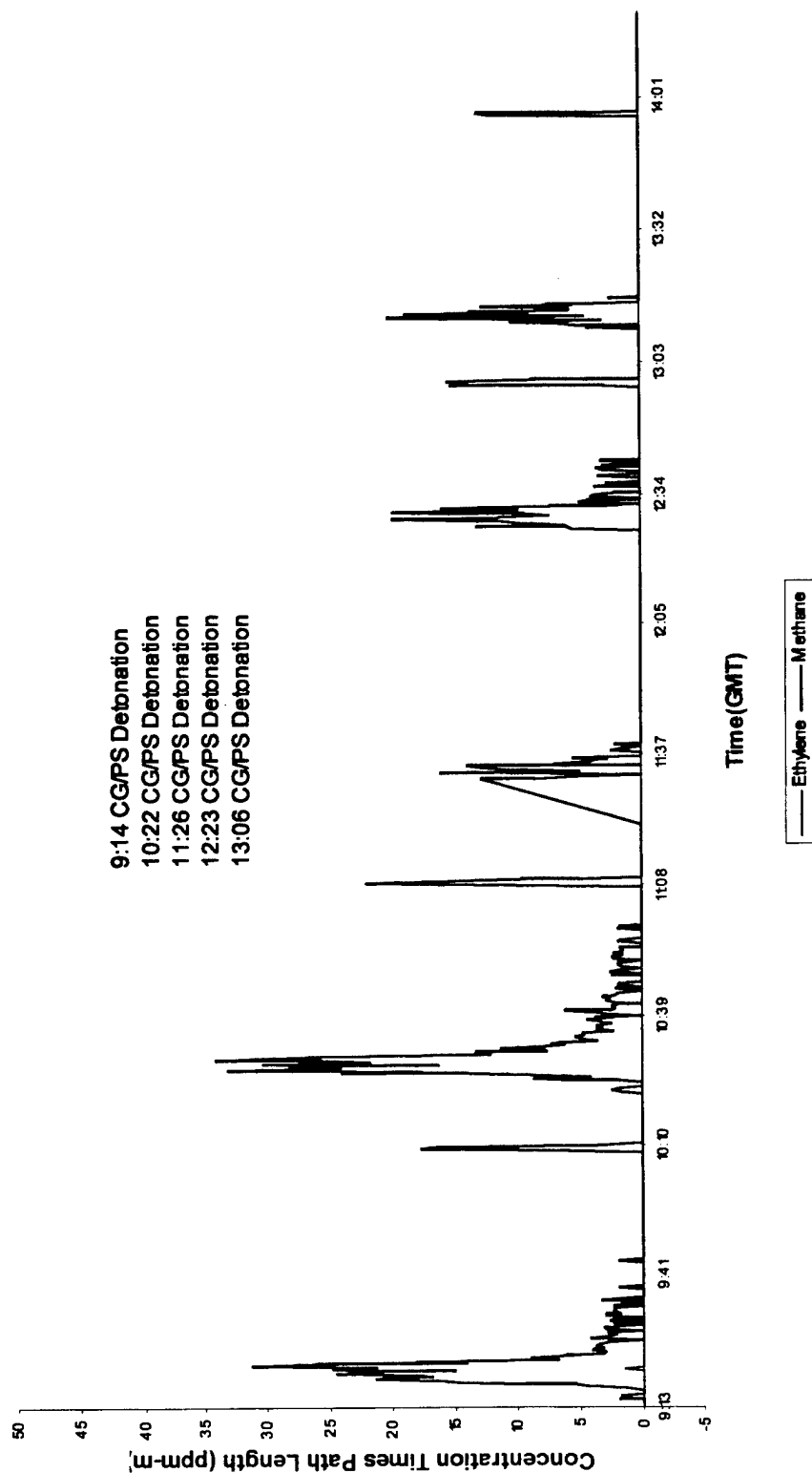


Figure 8.21

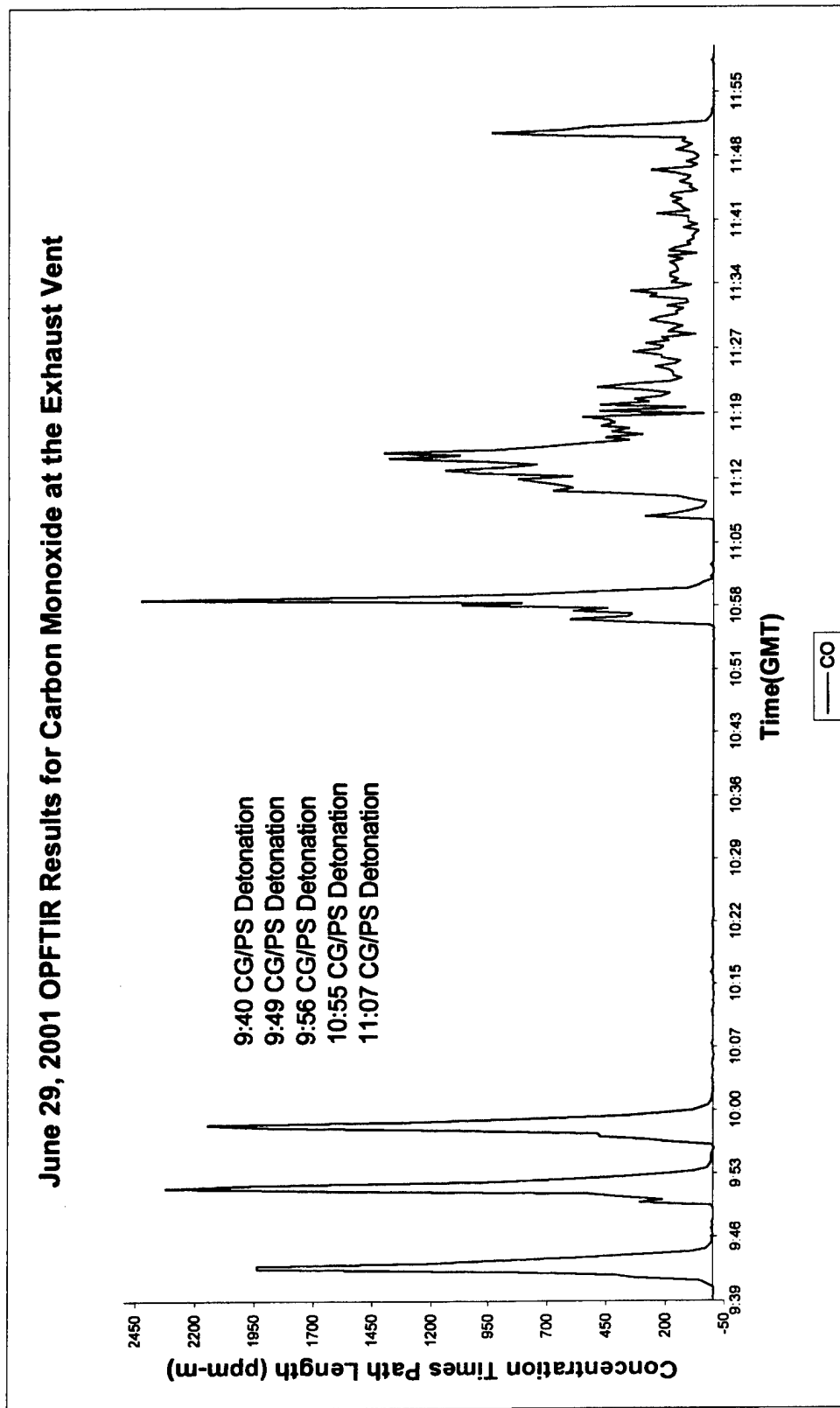


Figure 8.22

# June 29, 2001 OPFTIR Results for Acetylene, Ethylene, and Methane at the Exhaust Vent

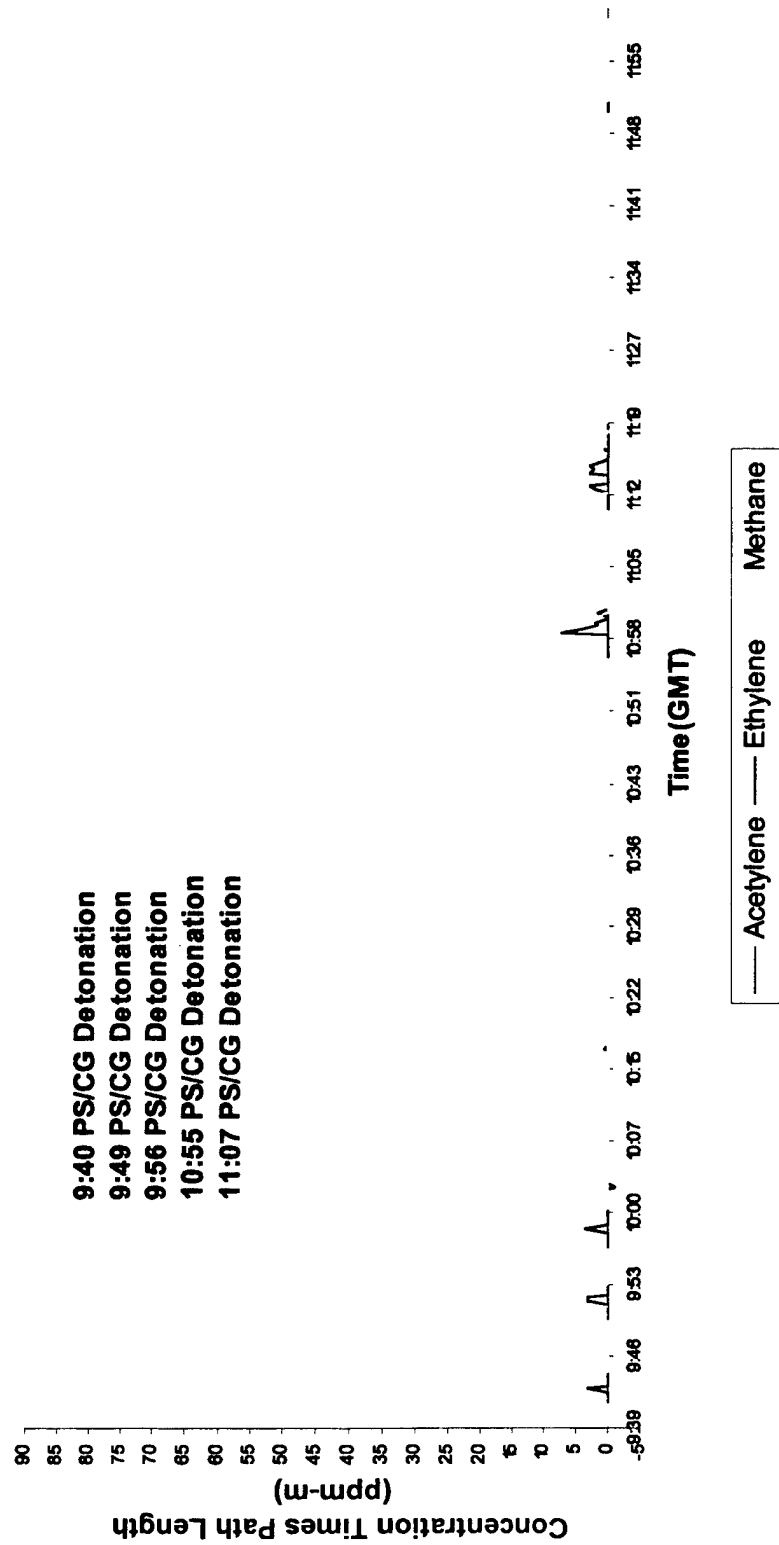


Figure 8.23

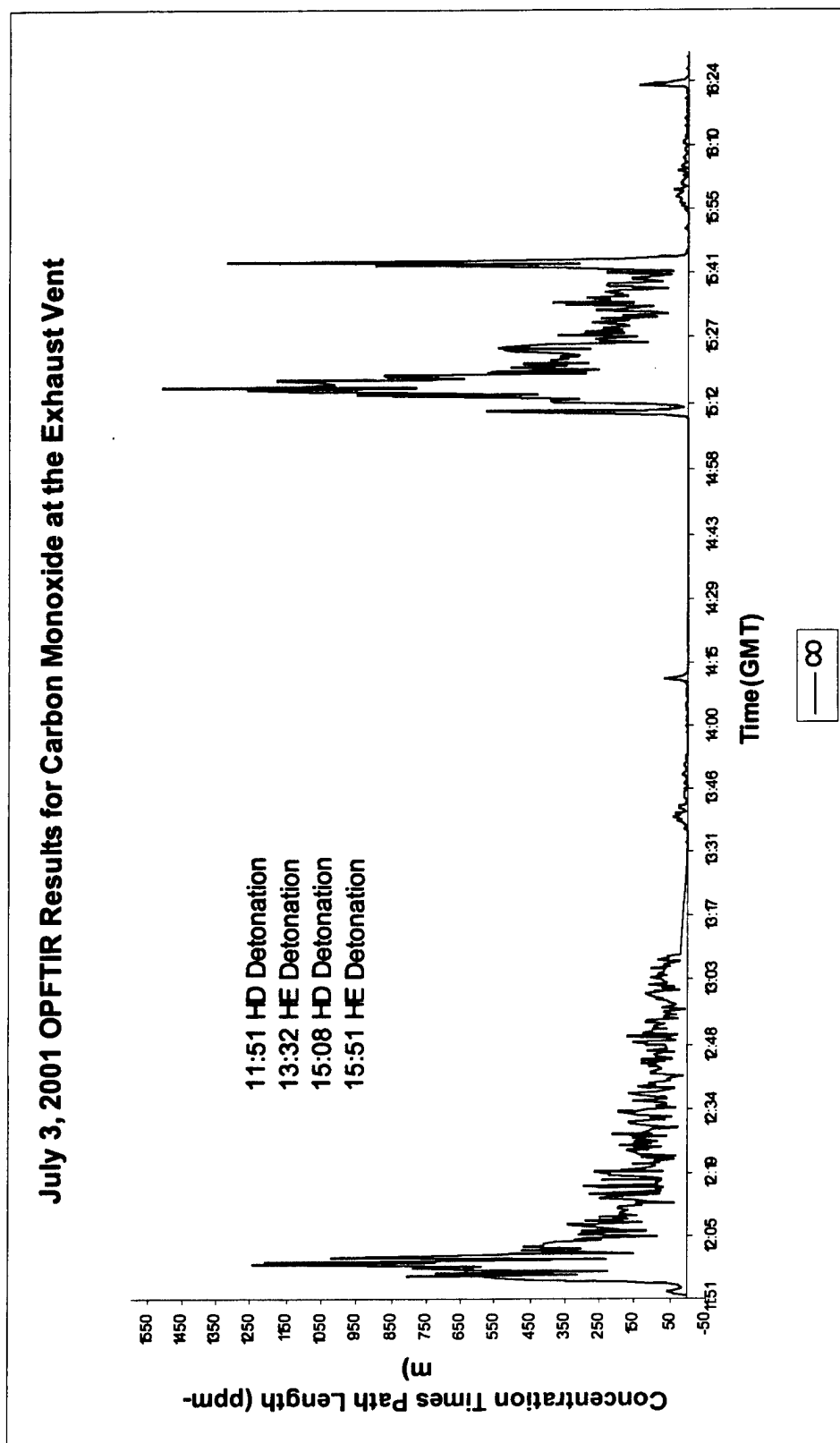


Figure 8.24

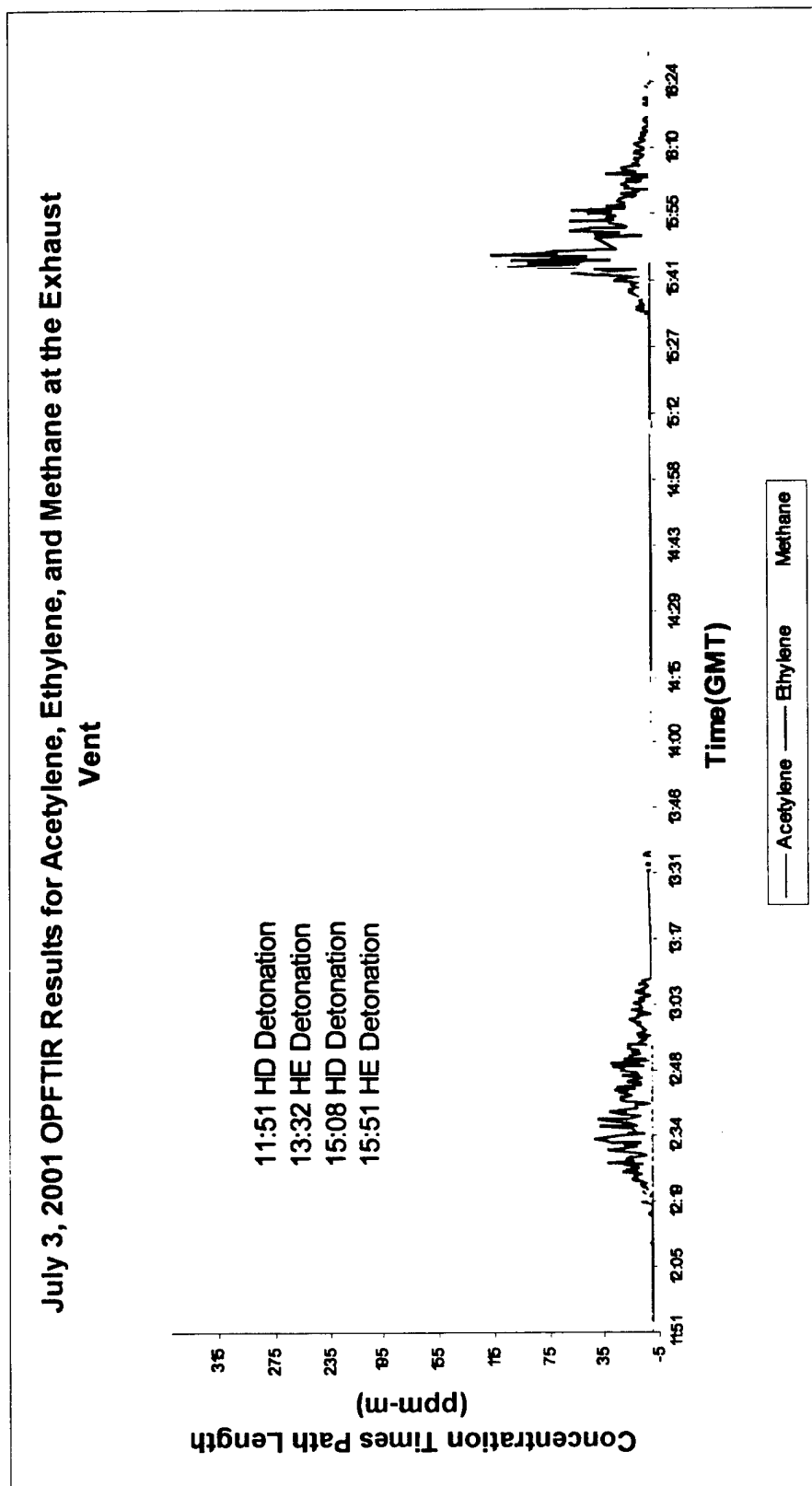




Figure 8.25

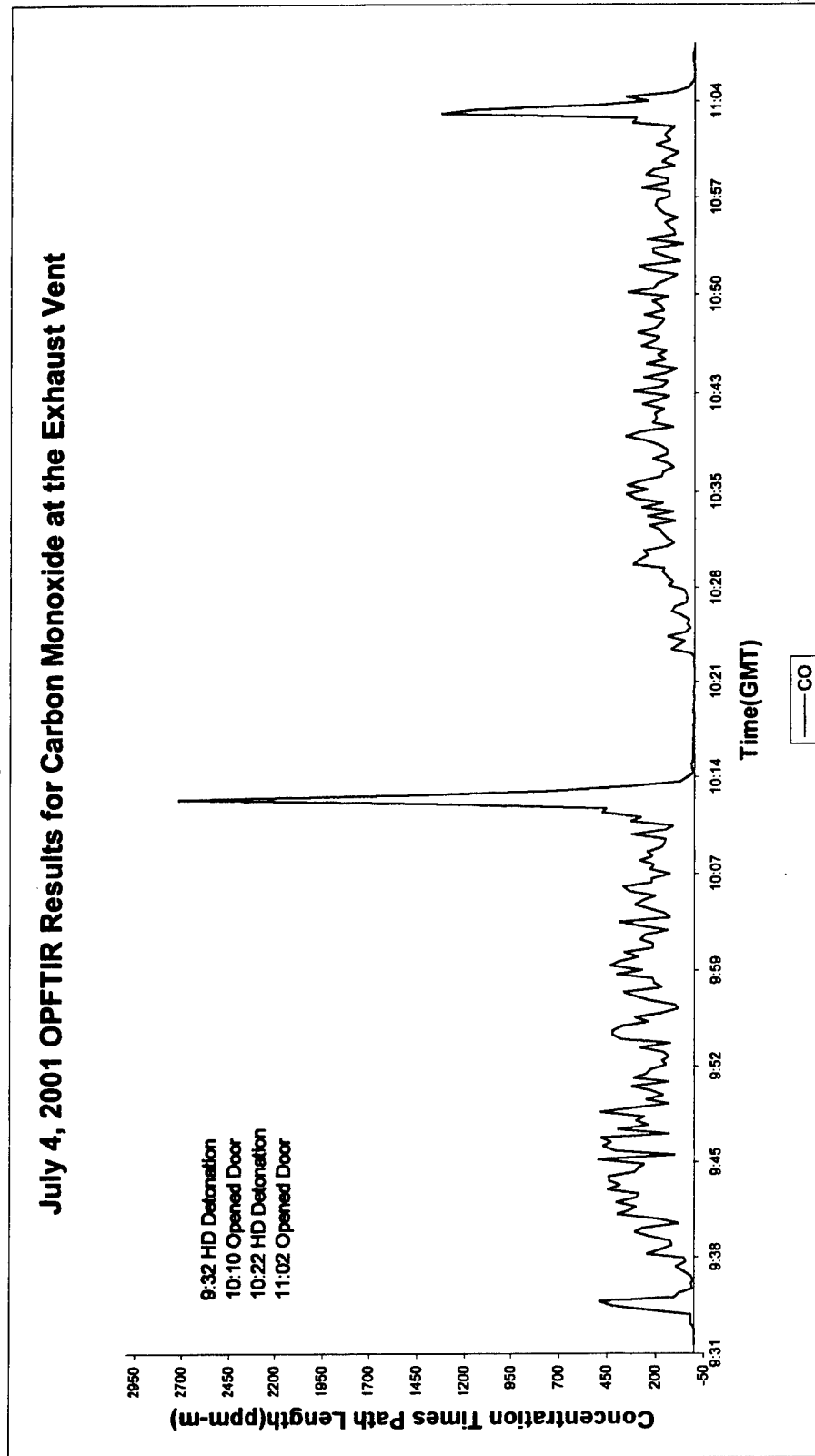


Figure 8.26

# July 4, 2001 OPFTIR Results for Acetylene, Ethylene, and Methane at the Exhaust Vent

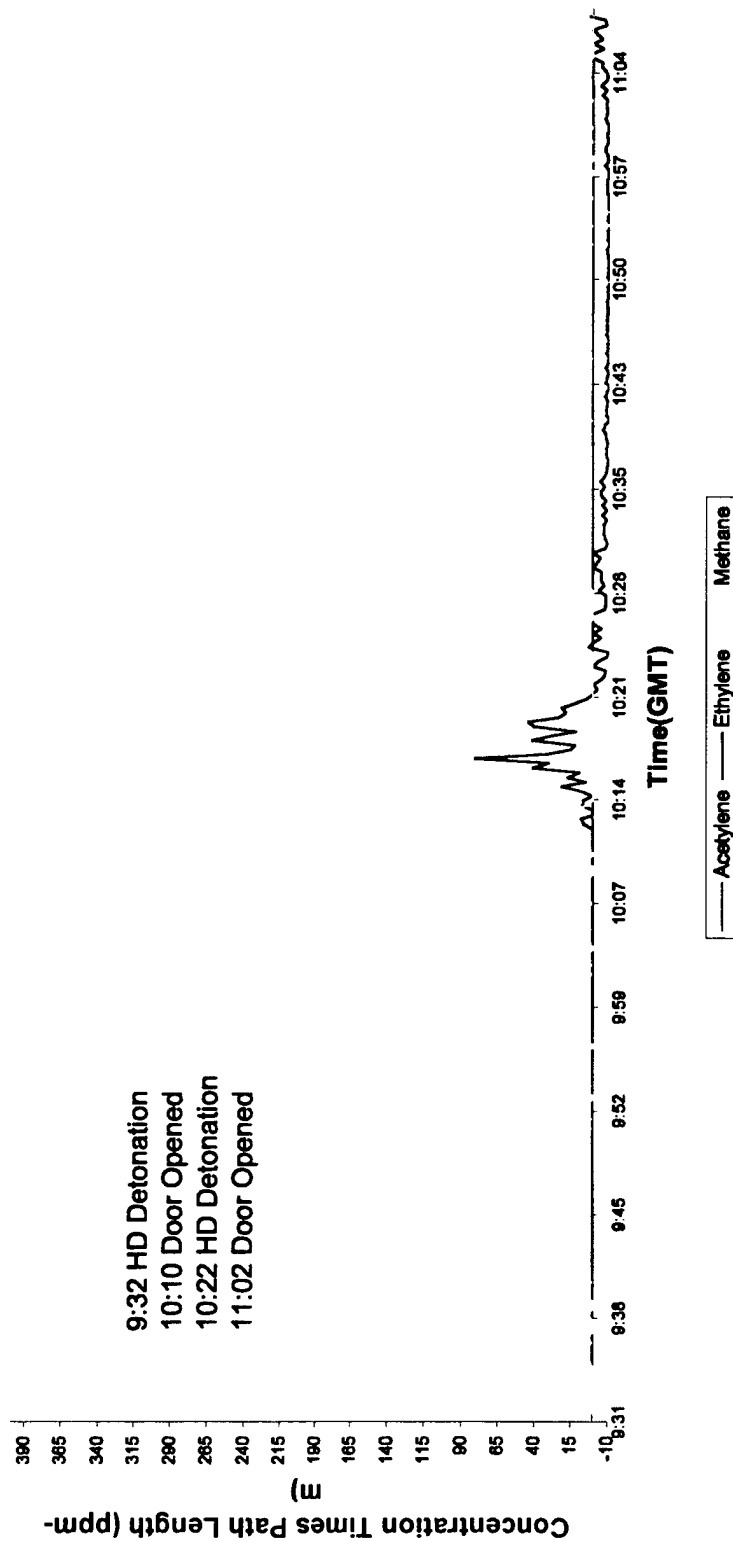


Figure 8.27

# July 5, 2001 OPFTIR Results for Carbon Monoxide at the Exhaust

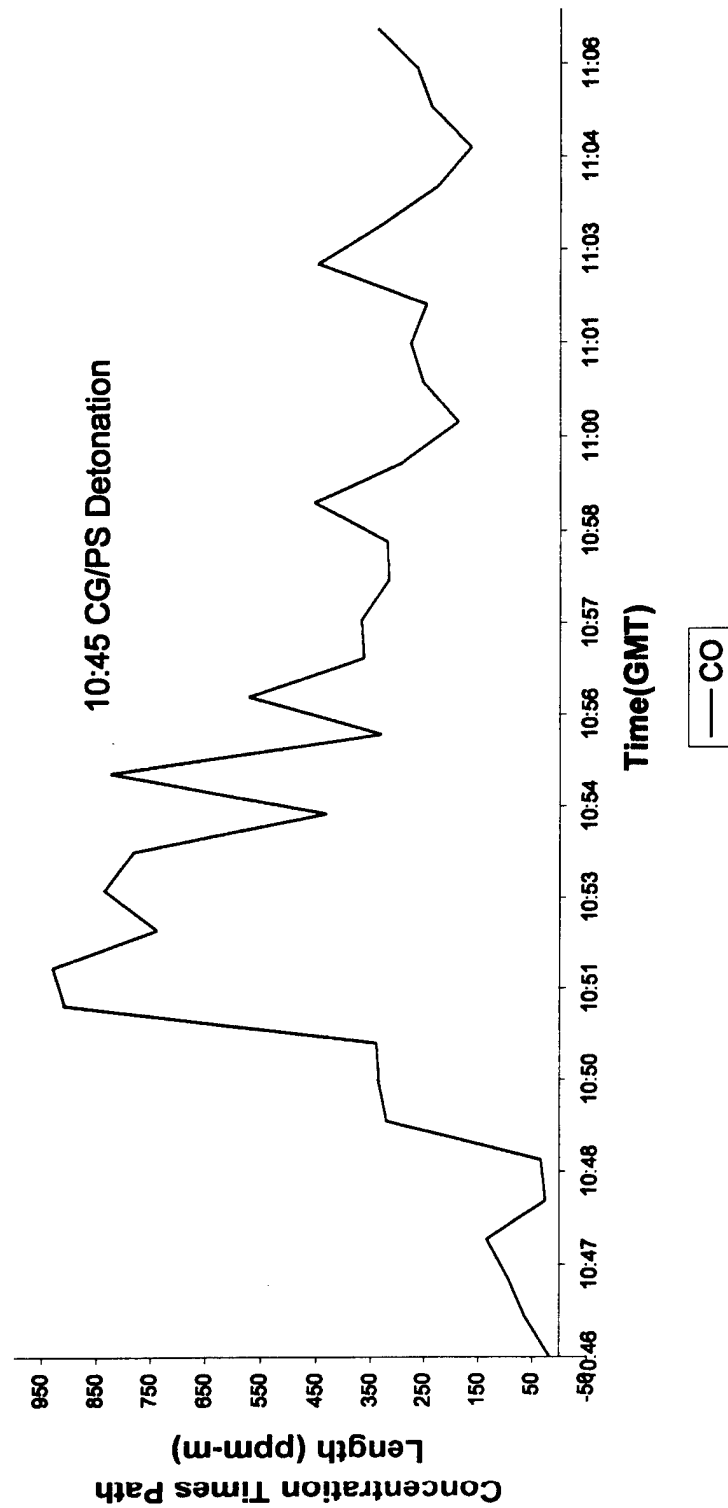
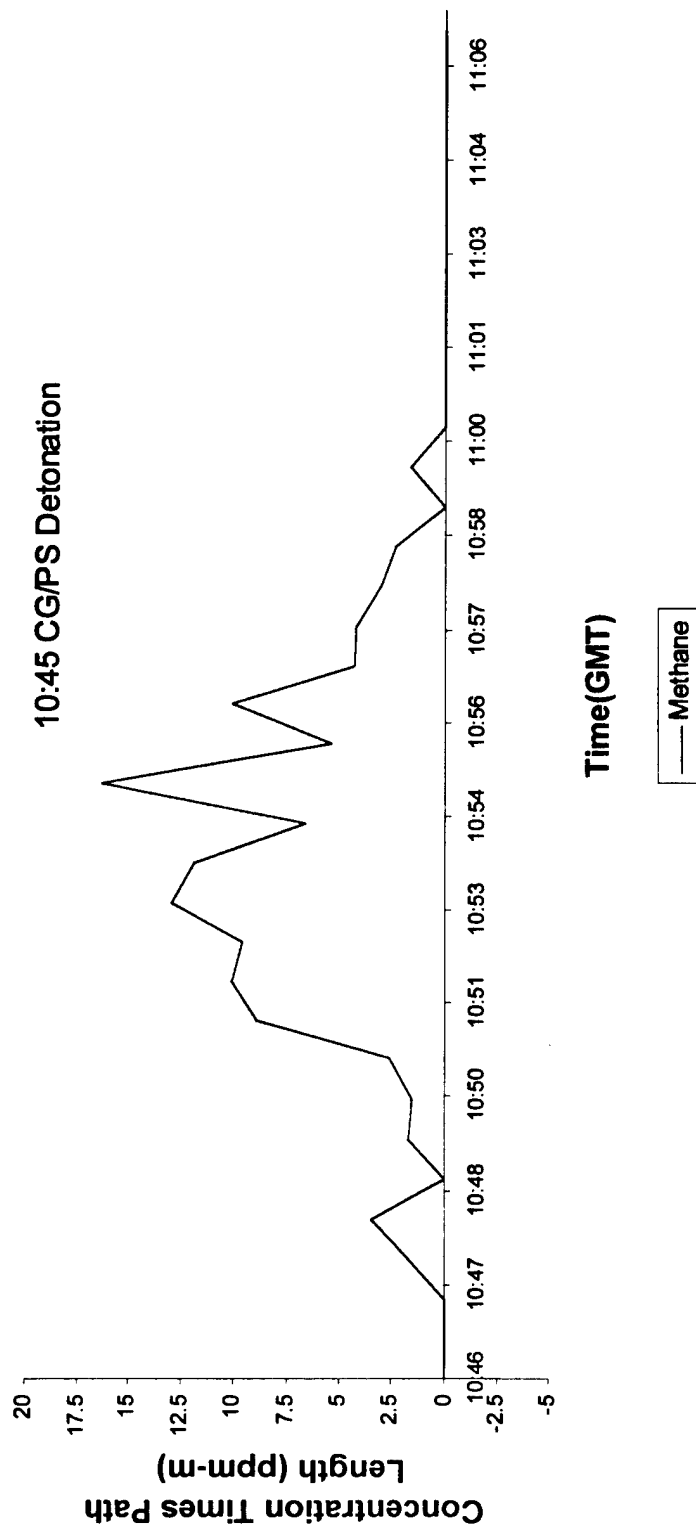


Figure 8.28

### July 5, 2001 OPFTIR Results for Methane at the Exhaust Vent



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**APPENDIX 9.**  
**DATA TABLES**

**Table 9.1. Physical Dimensions and Chemical Contents of WWI Munitions at Poelkapelle Which Do Not Contain Arsenic Compounds**

Type	Caliber	Dimensions (cm)		EXPLOSIVE MASS (g)						TOXIC (CHEMICAL AGENT) MASS (g)				
		Length	Wall	TNT/AN (60 / 40)	PIC	TNN	DNB	TNT/AC/AN (45/ 40/ 15)	PS	CG	CB	HD		
HE	7.7 cm	31	1.0	900	-	-	-	-	-	-	-	-		
				-	940	-	-	-	-	-	-	-		
				820	75	-	-	-	-	-	-	-	-	
				-	520	265	-	-	-	-	-	-	-	
				2000	-	-	-	-	-	-	-	-	-	
CG	10.5 cm	38	1.5	850	-	-	1150	-	-	-	-	-		
				-	2000	-	-	-	-	-	-	-	-	
				-	21 - 23	-	-	-	-	375-565	460- 610	-	-	
				-	55 - 63	-	-	-	-	-	1850 - 2150	-	-	
				-	-	-	-	-	-	258 - 308	1713	-	-	
HD	10.5 cm	38	1.5	-	55 - 63	-	-	-	-	-	-	-		
				25	?	?	?	?	?	?	750	-	-	
				31	-	21 - 23	-	-	-	-	-	74-148	650 - 760	
				-	-	-	-	-	-	-	-	-	-	
				-	-	-	-	-	-	-	-	-	-	

Caliber Abbreviations: L WM = Leichte W/ffmine

Explosive Abbreviations

TNT/AN = Trinitrotoluene (60%) / ammonium nitrate (40%)

PIC = Picric acid (also called 2, 4, 6 trinitrophenol)

TNN = Trinitronaphthalene

DNB = Dinitrobenzene

TNT/AC/AN = Trinitrotoluene (45%) / ammonium chloride (40%) / ammonium nitrate (15%)

Toxic (Chemical Agent) Abbreviations

CG = Phosgene (liquid)

HD = Mustard (semi-solid to liquid, 2 to 10% polymerization likely)

**Table 9.2. Physical Dimensions and Chemical Contents of Arsenic - Containing WWI Munitions at Poelkapelle**

Type	Cali ber	DIMENSIONS (cm)		EXPLOSIVE MASS (g)						TOXIC (CHEMICAL AGENT) MASS (g)				
		Length	Wall	TNT/AN (60 / 40)	TNT	TNT/TNN	PIC	TNN	DNB	TNT/AC/AN (45/40/15)	AsCl <sub>3</sub> /CG	As <sub>2</sub> O <sub>3</sub> / P/wax	Clark I	Clark II
HE + Smoke	7.7 cm	31	1.0	900	-	-	-	-	-	-	-	75	-	-
				-	-	-	940	-	-	-	-	75	-	-
				820	-	-	75	-	-	-	-	75	-	-
				-	-	-	520	265	-	-	-	75	-	-
				2000	-	-	-	-	-	-	-	150	-	-
Clark I	7.7 cm	31	1.0	850	-	-	-	-	1150	-	-	150	-	-
				-	620-675	-	-	-	-	-	-	-	105-142	-
Clark II	7.7 cm	31	1.0	-	-	1180-1330	-	-	-	-	-	-	105-142	-
				-	620-675	-	-	-	-	-	-	-	-	290-410
AsCl <sub>3</sub>	4.5 in	37	1.4	-	-	-	-	-	-	258-308	2688	-	-	-

**Explosive Abbreviations**

TNT/AN = Trinitrotoluene (60%) / ammonium nitrate (40%)  
TNT/TTN = Trinitrotoluene / trinitronaphthalene (ratio not specified)  
PIC = Picric acid (also called 2, 4, 6 trinitrophenol)  
TNN = Trinitronaphthalene  
DNB = Dinitrobenzene  
TNT/AC/AN = Trinitrotoluene (45%) / ammonium chloride (40%) / ammonium nitrate (15%)

**Toxic (Chemical Agent) Abbreviations**

AsCl<sub>3</sub> / CG = Arsenic trichloride / phosgene (thick, oily liquid)  
As<sub>2</sub>O<sub>3</sub> / P/wax = Arsenic trioxide / red phosphorus / wax (probably like a hard wax)  
Clark I = Diphenylchloroarsine (oily liquid)  
Clark II = Diphenylcyanoarsine (oily liquid)



**Table 9.3. Physical Properties of Chemical Agents and Explosives**

Symbol	Compound Name	Brut Formula	Mol. Wt	Melting Point (°C)	Boiling Point (°C)	Density (g/ml)	Vapor Pressure (mm Hg)	Comments
CG	Phosgene	$\text{COCl}_2$	99	-104	8	1.37	1180 mm @ 20 °C	Decomposes to HCl and $\text{CO}_2$ in contact with water
PS	Chloropicrin	$\text{CCl}_3\text{NO}_2$	164.38	-64.5	111.48	1.6566 @ 20 °C	25.3 mbar @ 20 °C	
HD	Mustard	$(\text{ClCH}_2\text{CH}_2)_2\text{S}$	159	14	228	1.2 @ 20 °C	0.1 mm @ 30 °C	Semi-soft solid to thick, oily liquid.
CB	Chlorobenzene	$\text{C}_6\text{H}_5\text{Cl}$	112.56	-45.6	132	1.1056 @ 20 °C	11.7 mbar @ 20 °C	
$\text{AsCl}_3$	Arsenic trichloride	$\text{AsCl}_3$	181	-16	130	2.2 @ 25 °C	10 mm @ 23 °C	Colorless, oily liquid. Vapor density is 6.3 g/cc @ 20 °C.
$\text{As}_2\text{O}_3$	Arsenic trioxide		396	278	460	4.2	-	1.8 g dissolves in 100 ml of water @ 20 °C
CLARK I	Diphenylchloroarsine	$(\text{C}_6\text{H}_5)_2\text{AsCl}$	265	44	333 (decomposes)	1.36	5E-05 mm @ 20 °C	Oily liquid to semi- solid. Vapor density is 9.15 g/cc @ 20 °C; Decontaminate surfaces with caustic soda or bleach.
CLARK II	Diphenylcyanoarsine	$(\text{C}_6\text{H}_5)_2\text{AsCN}$	258	31	213 (@ 21 mm Hg)	1.3 @ 20 °C	2E-04 @ 20 °C	Oily liquid to semi- solid. Vapor density is 8.8 g/cc @ 20 °C. Decontaminate surfaces with caustic soda or bleach.
WP	White phosphorus	$\text{P}_4$	31	44	280	1.8	1 mm @ 76 °C	Waxy solid. Vapor density is 4.4 g/cc @ 20 °C. Auto-ignites in contact with air at @ 30 °C.
TNT	Trinitrotoluene	$\text{C}_6\text{H}_5(\text{NO}_2)_3$	219	81	240	1.6	-	Solid
TNN	Trinitronaphthalene	$\text{C}_{10}\text{H}_7(\text{NO}_2)_3$	263	247	-	-	-	Solid
PIC	Picric acid	$\text{C}_6\text{H}_2(\text{NO}_2)_3\text{O}_H$	229	121	>300	1.7	-	Solid
DNB	Dinitrobenzene	$\text{C}_6\text{H}_4(\text{NO}_2)_2$	168	117	319	1.6	-	Solid
AC	Ammonium chloride	$\text{NH}_4\text{Cl}$	53	338	520	1.5	1 mm @ 160 °C	Solid
AN	Ammonium nitrate	$\text{NH}_4\text{NO}_3$	80	170	210 (decomposes)	1.7	-	Solid

NOTE: Picric acid is also called 2, 4, 6- trinitrophenol

**Table 9.4. Fill Characteristics of 77 MM LFKGR Shells**

Type	Agent	% of fill weight (g)
GelbKr	HD	80 to 90
	CB	10 to 20
GrünKr	PS	33-50
	CG	50 to 66
BlauKr	DA	105 to 142

Remark: total volume of fill is 0,67 Liter

**Table 9.5. Description of Sampling and Analysis Systems used in the Poelkapelle Test Phase I**

Method ID	Sample type	Analysis	Location Sampled	Lab responsible
A	Quartz Filter followed by three impingers containing water.	Quartz filter is weighted for total particulate matter and chloride Impinger contents is analyzed for chloride	Expansion Chamber, Exhaust Duct	RMA
B	Millipore particle filter followed by TENAX tube	Analyzed for total arsenic, aluminum, copper and lead		RMA
C	TENAX	Tube flash desorbed for CG, HD, DA, PS and chlorobenzene.	Expansion Chamber, Exhaust Duct	RMA
D	DAAMS	Tube flash desorbed for HD, breakdown products as appropriate.	Detonation Chamber, Expansion Chamber, Exhaust Duct, Perimeter	ECBC
E	MINICAMS	CG, HD, PS	Expansion Chamber, Exhaust Duct	ECBC
B	Wipe samples <sup>1</sup>	Analysis for As	Detonation Chamber	RMA
C	Pea gravel samples <sup>2</sup>	Analysis for residual CWA	Detonation Chamber	RMA
G	FTIR	DA, DC, CO, CG, HD, PS, Methane, Ethylene, Acetylene	Above Detonation Chamber Door, Exhaust Vent	ECBC

<sup>1</sup> some wipes also analysed for residual explosives, DA or HD

<sup>2</sup> Not systematically taken during phase I.

**Table 9.6. Description of Sampling and Analysis Systems used in the Poelkappelle Test Phase II**

Method ID	Sample type	Analysis	Location Sampled	Lab responsible
B	Millipore particle filter followed by TENAX tube	Analyzed for total arsenic, aluminum, copper and lead	Detonation Chamber	RMA
C	TENAX	Tube flash desorbed for CG, HD, DA, PS, and chlorobenzene.	Detonation Chamber	RMA
D	DAAMS	Tube flash desorbed for HD and degradation products	Detonation Chamber, Expansion Chamber, Exhaust Duct, Exhaust Vent, Perimeter	ECBC
E	MINICAMS	PS, CG, HD	Expansion Chamber, Exhaust Duct, Exhaust Vent	ECBC
C	Wipe samples	Analysis for residual CWA	Detonation Chamber, Expansion Chamber, Bag House Filter	RMA
B	Wipe samples	Analyzed for total arsenic, aluminum, copper, and lead	Detonation Chamber, Expansion Chamber, Bag House Filter	
C	Pea gravel samples	Analysis for residual CWA	Detonation Chamber	RMA
B	Pea gravel samples	Analyzed for total arsenic, aluminum, copper, and lead	Detonation Chamber	RMA
G	FTIR	DA, DC, CO, CG, HD, PS, Methane, Ethylene, Acetylene	Exhaust Vent	ECBC
H	Charcoal adsorption tube	Volatile (organic) aromatics	Expansion Chamber, Exhaust Vent, Exhaust Duct	RMA
F	Charcoal adsorption tube (ORBO52)	CG specific	Expansion Chamber, Exhaust Vent, Exhaust Duct	RMA

**Table 9.7. Sample Collection: Approximate Flow Rates and Sampling Times**

SAMPLE TYPE	FLOW RATE (L/MIN)	SAMPLING TIME at (minutes, unless stated otherwise)	METHOD CODE (see Appendix for methods)
42 mm Millipore	1.0	10 to 20	B
TENAX tubes	0.05	20 to 60	C
Bubbler train	1.5	20 to 30	A
Charcoal tubes (ORBO)	0.2	20 to 30	F,H

**Table 9.8. Overview of Detonations in the CDC**

Order of detonation	Date	Type <sup>(1)</sup>	Idf number	donor explosive and additives
1	14May	na	na	TNT
2	14May	HE	na	RDX, 2 bags H2O
3	15May	CG	2652	RDX, 3 lbs; 2 bags H2O; 2 bags H2O2
4	15May	CG	2664	RDX, 4 lbs; 2 bags H2O; 2 bags H2O2
5	15May	CG	2655	RDX, 4 lbs; 2 bags H2O; 2 bags H2O2
6	16May	HD	2797	RDX, 4 lbs; 4 bags H2O
7	16May	HD	2812	RDX, 4 lbs; 2 bags H2O; 2 bags H2O2
8	16May	na	na	IREMITE, 4 lbs
9	16May	HD	2795	RDX, 3 lbs; AN, 1.3 lbs; 4 bags H2O;
10	16May	na	na	TNT
11	17May	DA	3166	RDX, 2.5 lbs; 4 bags H2O
12	17May	DA	3167	RDX, 2.5 lbs; 2 bags H2O; 2 bags H2O2
13	17May	DA	3168	RDX, 1.5 lbs; AN, 1 lb; 4 bags H2O;
14	18May	CG	3102	RDX, 4 lbs; 4 bags H2O
15	18May	CG	3117	RDX, 3 lbs; AN, 1 lb; 4 bags H2O;
16	21May	DA	3151	RDX, 2.5 lbs; 4 bags H2O
17	21May	HD	2817	RDX, 4 lbs; 4 bags H2O
18	12June	DA	3165	RDX, 2 lbs; 4 bags H2O
19	12June	DA	3152	DBS, 2 lbs; 4 bags of H2O
20	12June	DA	3199	RDX, 2 lbs; 4 bags H2O
21	12June	DA	3196	DBS, 2 lbs; 4 bags of H2O
22	13June	DA	3193	DBS, 2 lbs; 2 bags of H2O; 2bags of Al2O3
23	13June	DA	3194	DBS, 2 lbs; 2 bags of H2O; 2 bags of Al2O3
24	13June	DA	3183	DBS 2 lbs, 4 bags of H2O; 2 bags of Al2O3
25	13June	DA	3184	DBS, 2 lbs; 4 bags of H2O; 2 bags of Al2O3
26	14June	DA	3185	DBS, 2 lbs; 4 bags of H2O; 2 bags of Kaolinite
27	14June	DA	3176	DBS, 2 lbs; 4 bags of H2O; 2 bags of Kaolinite
28	14June	DA	3182	DBS, 2 lbs; 4 bags of H2O; 2 bags of Silica gel
29	14June	DA	3164	DBS, 2 lbs; 2 bags of H2O.; 2 bags of H2O2; 2 bags of Al2O3
30-40	15June	DA	3172; 3221; 3220; 3212; 3223; 3205; 3201; 3206; 3208; 3219; 3243;	DBS, 2 lbs; 4 bags of H2O;
41-44	15June	DA	3242; 3293; 3237; 3235	DBS, 2 lbs; 4 bags of H2O;
45	20June	HD	2665	DBS, 4 lbs; 5 bags H2O, 2 bags Al2O3
46	20June	HD	2788	DBS, 4 lbs; 5 bags H2O, 2 bags Al2O3
47	20June	HD	2659	DBS, 4 lbs; 4 bags H2O, 2 bags Al2O3
48	20June	HD	2662	DBS, 4 lbs; 4 bags H2O, 2 bags Al2O3
49	21June	HD	2780	DBS, 4 lbs; 6 bags H2O, 2 bags Al2O6
50	21June	HD	2807	DBS, 4 lbs; 6 bags H2O
51	21June	HD	3103	DBS, 4 lbs; 8 bags H2O

(1) all types were GE 77mm LFKGr

**Table 9.8. Overview of Detonations in the CDC (continued)**

Order of detonation	Date	Type <sup>(1)</sup>	idf number	donor explosive and additives
52	21June	HD	3104	DBS, 4 lbs; 10 bags H2O
53	22June	HD	3106	DBS, 4 lbs; 10 bags H2O
54	22June	HD	3135	DBS, 4 lbs; 8 bags H2O, 2 bags H2O2
55	22June	na	na	DBS, 1.5 lbs; PVC pipe
56	25June	na	na	DBS, 4 lbs
57	26June	na	na	RDX, 4 lbs
58	27June	CG	3121	DBS, 4 lbs 4 bags H2O, 2 bags Al2O3
59	27June	CG	3082	4 lbs
60	27June	CG	3079	DBS, 4 lbs; 6 bags H2O, 2 bags Al2O3
61	27June	CG	3078	DBS, 4 lbs; 6 bags H2O, 2 bags Al2O3
62	27June	CG	2983	DBS, 4 lbs; 6 bags H2O, 2 bags Al2O3
63	27June	CG	2977	DBS, 4 lbs; 8 bags H2O, 2 bags Al2O3
64	28June	CG	3139	DBS, 4 lbs; 8 bags H2O, 2 bags Al2O3
65	28June	CG	2976	DBS, 4 lbs; 4 bags H2O
66	28June	CG	2980	DBS, 4 lbs; 4 bags H2O
67	28June	CG	2799	DBS, 4 lbs; 4 bags H2O
68	28June	CG	2603	DBS, 4 lbs; 4 bags of H2O,
69-73	29June	CG	2975; 3019; 3017; 3021; 2649	DBS, 4 lbs; 4 bags of H2O,
74	3July	HD	3043	DBS, 4 lbs; 4 bags of H2O, 2 bags bags of Al2O3
75	3July	HE	na	DBS, 4 lbs; 4 bags of H2O,
76	3July	HD	3033	DBS, 4 lbs; 4 bags H2O, 2 bags Al2O4
77	3July	HE	na	DBS, 4 lbs; 4 bags H2O
78	4July	HD	3132	DBS, 4 lbs; 4 bags H2O, 2 bags Al2O3
79	4July	HD	3056	DBS, 4 lbs; 4 bags H2O, 2 bags Al2O3
80	5July	CG	3111	DBS, 4 lbs; 4 bags H2O, 2 bags Al2O3
81	5July	DA	3318	DBS(+RDX), 4 bags of H2O; BX-24, 1 kg;
82	5July	HE	na	RDX
83	5July	DA	3317	DBS(+RDX), 4 bags of H2O; BX-24, 1 kg;
84	5July	HE	na	RDX
85-89	9July	DA	3229; 3232; 3239; 3240; not_registered	DBS(+RDX), 4 bags of H2O;
90	9July	HE	na	RDX

(1) all types were GE 77mm LFKGr

**Table 9.9. Phase I. Airborne Concentrations of Phosgene and Chloropicrin in Expansion Chamber (EC) and Duct after Detonation of Gruenkreuz Shells**

				PS			CG				
Order of detonation	Date	Shell	Donor Explosive and Additives	EC (mg/m³)	EC (mg/m³)	Duct (mg/m³)	EC (mg/m³)	EC (mg/m³)	Duct (mg/m³)	EC (mg/m³)	Duct (mg/m³)
3	15-May	2652	RDX, 3 lbs; 2 bags H2O; 2 bags H2O2	142	777	40.8	44.6	245	14.0		
4	15-May	2664	RDX, 4 lbs; 2 bags H2O; 2 bags H2O2	22.68	86.22	4.98	8.00	30.05	2.97		
5	15-May	2655	RDX, 4 lbs; 2 bags H2O; 2 bags H2O2	18.84	75.16	0.70	6.83	26.73	1.73		
14	18-May	3102	RDX, 4 lbs; 4 bags H2O	ND	ND	ND	1.22	8.48	ND		
15	18-May	3117	RDX, 3 lbs; AN, 1 lb; 4 bags H2O;	2.05	ND	ND	2.45	7.67	2.38		

The detection limit for particulate matter is 0.002 g/m<sup>3</sup>

**Table 9.10. Concentration of Chloride and Particulate Matter in the Expansion Chamber (EC), Duct after Detonating Phosgene/Chloropicrin Shells (Gruenkreuz)**

Order of detonation	Date	Shell	Donor Explosive and Additives	Chloride						Particulate Matter	
				Volatile		Solid		EC (g/m <sup>3</sup> )	Duct (g/m <sup>3</sup> )	EC (g/m <sup>3</sup> )	Duct (g/m <sup>3</sup> )
				EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )				
3	15-May	2652	RDX, 3 lbs; 2 bags H2O; 2 bags H2O2	8.4	3.02	1970	ND	5.02	ND		ND
4	15-May	2664	RDX, 4 lbs; 2 bags H2O; 2 bags H2O2	3710	ND	21.63	26.11	0.30			ND
5	15-May	2655	RDX, 4 lbs; 2 bags H2O; 2 bags H2O2	1260	ND	41.56	2.80	11.60			ND
14	18-May	3102	RDX, 4 lbs; 4 bags H2O	140	ND	24.97	1.28	NA		NA	
15	18-May	3117	RDX, 3 lbs; AN, 1 lb; 4 bags H2O;	14.0	ND	94.00	4.00	0.80			0.10



**Table 9.11. Phase I. Airborne Concentrations of Chlorobenzene (CB) and Mustard (HD) in Expansion Chamber (EC) and Duct after Detonating Mustard Shells**

Order of detonation	Date	Shell	Donor Explosive and Additives	CB			HD		
				EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	EC (mg/m <sup>3</sup> )	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )
6	16-May	2797	RDX, 4 lbs; 4 bags H2O	28,80	91,88	48,49	11,11	6,13	1,09
7	16-May	2812	RDX, 4 lbs; 2 bags H2O; 2 bags H2O2	37,58	Overload	26,94	16,26	9,79	1,14
9	16-May	2795	RDX, 3 lbs; AN, 1.3 lbs; 4 bags H2O;	38,56	Overload	5,17	8,54	7,40	0,88
17	21-May	2817	RDX, 4 lbs; 4 bags H2O	ND	ND	ND	ND	ND	ND

**Table 9.12. Phase I. Concentrations of Chloride and Particulate Matter in the Expansion Chamber (EC) and Duct after Detonating Mustard Shells**

				Chloride						Particulate Matter	
				Volatile		Solid					
Order of detonation	Date	Shell	Donor Explosive and Additives	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	EC (g/m <sup>3</sup> )	Duct (g/m <sup>3</sup> )		
6	16-May	2797	RDX, 4 lbs; 4 bags H2O	ND	ND	209	NA	0.54	ND		
7	16-May	2812	RDX, 4 lbs; 2 bags H2O; 2 bags H2O2	42.5	ND	167	NA	4.85	ND		
9	16-May	2795	RDX, 3 lbs; AN, 1.3 lbs; 4 bags H2O;	2.10	1.43	28.5	NA	NA	NA		
17	21-May	2817	RDX, 4 lbs; 4 bags H2O	0.01	13.5	90.0	NA	0.1	ND		

**Table 9.13. Phase I. Airborne Concentrations of DA, Concentrations of Chloride, and Particulate Matter in the Expansion Chamber (EC) and Duct after Detonating "Blue Cross" Shells Containing DA**

Order of cetonation	Date	Shell	Donor Explosive and Additives	DA			Chloride				Particulate Matter	
							Volatile		Solid			
				EC (mg/m <sup>3</sup> )	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	EC (mg/m <sup>3</sup> )	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	EC (g/m <sup>3</sup> )	Duct (g/m <sup>3</sup> )	
11	17-May	3166	RDX, 2.5 lbs; 4 bags H2O	4.58	ND	ND	ND	ND	9.00	4.64	NA	NA
12	17-May	3167	RDX, 2.5 lbs; 2 bags H2O; 2 bags H2O2	10.96	2.53	ND	ND	ND	11.73	8.33	NA	NA
13	17-May	3168	RDX, 1.5 lbs; AN, 1 lb; 4 bags H2O;	3.89	ND	ND	ND	ND	16.78	ND	0.40	ND
16	21-May	3151	RDX, 2.5 lbs; 4 bags H2O	ND	ND	ND	0.02	0.02	17.08	ND	0.12	ND

**Table 9.14. Phase I. Concentrations of Chloropicrin in Expansion Chamber (EC), Duct (Airborne) and Detonation Chamber (DC) (Wall and Pea Gravel)**

Order of detonation	Date	Shell	Donor Explosive and Additives	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	Wall DC (mg/m <sup>2</sup> )	Pea Gravel (mg/kg)
58	27-June	3121	DBS, 4 bags H2O, 2 bags Al2O3	0.090	37.0	2.43	ND
59	27-June	3082	DBS, 4 bags H2O, 2 bags Al2O3	0.101	16.2	0.59	ND
60	27-June	3079	DBS, 6 bags H2O, 2 bags Al2O3	75.5	68.1	ND	ND
61	27-June	3078	DBS, 6 bags H2O, 2 bags Al2O3	0.167	0.1	ND	ND
62	27-June	2983	DBS, 6 bags H2O, 2 bags Al2O3	0.164	1.2	1.00	ND
63	27-June	2977	DBS, 8 bags H2O, 2 bags Al2O3	0.195	1.3	ND	ND
64	28-June	3139	DBS, 8 bags H2O, 2 bags Al2O3	ND	35.7	ND	ND
65	28-June	2976	DBS, 4 bags H2O	0.234	32.4	0.52	ND
66	28-June	2980	DBS, 4 bags H2O	0.263	94.9	ND	ND
67	28-June	2799	DBS, 4 bags H2O	0.241	39.2	6.22	ND
68	28-June	2603	DBS, 4 bags of H2O,	ND	25.9	0.77	ND
69-73	29-June	5_shell_series	DBS, 4 bags of H2O,	0.126	27.2	ND	ND
80	5-July	3111	DBS, 4 bags H2O, 2 bags Al2O4	ND	ND	N/A	N/A

**Table 9.15. Phase II. Concentrations of Phosgene in the Expansion Chamber (EC) (Airborne), Duct (Airborne) and Detonation Chamber (DC) (Wall and Pea Gravel)**

Order of detonation	Date	Shell	Donor Explosive and Additives	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	Wall DC (mg/m <sup>2</sup> )	Pea Gravel (mg/kg)
58	27-June	3121	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0.005	0.065	ND	ND
59	27-June	3082	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0.099	0.022	ND	ND
60	27-June	3079	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0.004	0.002	ND	ND
61	27-June	3078	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0.036	0.075	ND	ND
62	27-June	2983	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0.049	0.106	ND	ND
63	27-June	2977	DBS, 8 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0.022	0.046	ND	ND
64	28-June	3139	DBS, 8 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0.015	0.035	ND	ND
65	28-June	2976	DBS, 4 bags H <sub>2</sub> O	0.025	0.435	ND	ND
66	28-June	2980	DBS, 4 bags H <sub>2</sub> O	0.036	0.050	ND	ND
67	28-June	2799	DBS, 4 bags H <sub>2</sub> O	6.292	7.146	ND	ND
68	28-June	2603	DBS, 4 bags of H <sub>2</sub> O,	0.029	0.037	ND	ND
69-73	29-June	5_shell_series	DBS, 4 bags of H <sub>2</sub> O,	0.006	0.082	ND	ND
80	5-July	3111	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>4</sub>	0.004	0.142	ND	ND

**Table 9.16. Phase II. Concentrations of Diphsogene in Expansion Chamber (EC) (Airborne), Duct (Airborne) and Detonation Chamber (DC)(Wall and Pea Gravel)**

Order of detonation	Date	Shell	Donor Explosive and Additives	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	Wall DC (mg/m <sup>2</sup> )	Pea Gravel (mg/kg)
58	27-June	3121	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	ND	ND	ND
59	27-June	3082	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	ND	ND	ND
60	27-June	3079	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	ND	ND	ND
61	27-June	3078	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	ND	ND	ND
62	27-June	2983	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	ND	ND	ND
63	27-June	2977	DBS, 8 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	ND	ND	ND
64	28-June	3139	DBS, 8 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	ND	ND	ND
65	28-June	2976	DBS, 4 bags H <sub>2</sub> O	ND	ND	ND	ND
66	28-June	2980	DBS, 4 bags H <sub>2</sub> O	ND	ND	ND	ND
67	28-June	2799	DBS, 4 bags H <sub>2</sub> O	ND	ND	ND	57.0
68	28-June	2603	DBS, 4 bags of H <sub>2</sub> O,	ND	ND	ND	18.5
69-73	29-June	5_shell_series	DBS, 4 bags of H <sub>2</sub> O,	ND	ND	ND	ND
80	5-July	3111	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>4</sub>	ND	ND	N/A	N/A

**Table 9.17. Phase II. Concentrations of Chloropicrin in Expansion Chamber (EC) (Airborne), Duct (Airborne) and Detonation Chamber (DC) (Wall and Pea Gravel)**

Order of detonation	Date	Shell	Donor Explosive and Additives	EC (mg/m <sup>3</sup> )	Duct (mg/m <sup>3</sup> )	Wall DC (mg/m <sup>2</sup> )	Pea Gravel (mg/kg)
58	27 June	3121	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0,090	37,0	2,43	ND
59	27 June	3082	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0,101	16,2	0,59	ND
60	27 June	3079	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	75,5	68,1	ND	ND
61	27 June	3078	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0,167	0,1	ND	ND
62	27 June	2983	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0,164	1,2	1,00	ND
63	27 June	2977	DBS, 8 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	0,195	1,3	ND	ND
64	28 June	3139	DBS, 8 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	35,7	ND	ND
65	28 June	2976	DBS, 4 bags H <sub>2</sub> O	0,234	32,4	0,52	ND
66	28 June	2980	DBS, 4 bags H <sub>2</sub> O	0,263	94,9	ND	ND
67	28 June	2799	DBS, 4 bags H <sub>2</sub> O	0,241	39,2	6,22	ND
68	28 June	2603	DBS, 4 bags of H <sub>2</sub> O,	ND	25,9	0,77	ND
69-73	29 June	5_shell_series	DBS, 4 bags of H <sub>2</sub> O,	0,126	27,2	ND	ND
80	5 July	3111	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>4</sub>	ND	ND	N/A	N/A

**Table 9.18. Phase II. Concentrations of Volatile Aromatic Hydrocarbons (VAC)  
and Metals in the Expansion Chamber (Airborne);  
Residual Concentrations of Metals in Detonation Chamber (Pea Gravel)<sup>3</sup> for Detonations of Phosgene Shells**

						Heavy Metals									
				VAC			As		Al		Cu		Pb		
Order of detonation	Date	Shell	Donor Explosive and Additives	Benzene (mg/m <sup>3</sup> )	Toluene (mg/m <sup>3</sup> )	Xylene (mg/m <sup>3</sup> )	As airborne EC (mg/m <sup>3</sup> )	Pea gravel (mg/kg)	Al airborne EC (µg/cum)	Pea gravel (mg/kg)	Cu airborne EC (µg/cum)	Pea gravel (mg/kg)	Pb airborne EC (µg/cum)	Pea gravel (mg/kg)	
58	27-Jun	3121	DBS, 4 bags H2O, 2 bagsAl2O3	N/A	N/A	N/A	ND	N/A	5.14	N/A	1.25	N/A	ND	N/A	
59	27-Jun	3082	DBS, 4 bags H2O, 2 bagsAl2O3	N/A	N/A	N/A	ND	N/A	2.01	N/A	1.38	N/A	ND	N/A	
60	27-Jun	3079	DBS, 6 bags H2O, 2 bagsAl2O3	N/A	N/A	N/A	ND	N/A	1.57	N/A	0.39	N/A	ND	N/A	
61	27-Jun	3078	DBS, 6 bags H2O, 2 bagsAl2O3	N/A	N/A	N/A	ND	N/A	2.42	N/A	1.95	N/A	ND	N/A	
62	27-Jun	2983	DBS, 6 bags H2O, 2 bagsAl2O3	N/A	N/A	N/A	0.05	N/A	2.25	N/A	1.26	N/A	ND	N/A	
63	27-Jun	2977	DBS, 8 bags H2O, 2 bagsAl2O3	0.08	0.11	ND	ND	N/A	2.16	N/A	0.76	N/A	ND	N/A	
64	28-Jun	3139	DBS, 8 bags H2O, 2 bags Al2O3	0.41	0.28	ND	ND	ND	1.74	14758	0.40	642	ND	123	
65	28-Jun	2976	DBS, 4 bags H2O	0.39	0.33	ND	ND	ND	2.06	13880	0.77	569.6	ND	87	
66	28-Jun	2980	DBS, 4 bags H2O	0.41	0.27	ND	ND	ND	2.95	15844	1.00	1440.6	ND	270	
67	28-Jun	2799	DBS, 4 bags H2O	0.39	0.21	ND	ND	ND	3.36	13687	0.79	734	ND	131	
68	28-Jun	2603	DBS, 4 bags of H2O,	0.39	0.24	ND	ND	ND	3.35	17284	0.74	548	ND	77	
69-73	29-Jun	5_shell_series	DBS, 4 bags of H2O,	N/A	N/A	N/A	ND	ND	2.93	24077	2.17	1039	ND	118	
80	5-Jul	3111	DBS, 4 bags H2O, 2 bagsAl2O4	2.87	6.41	ND	ND	ND	1.02	N/A	0.46	N/A	ND	N/A	

<sup>3</sup>Notes: ND=not detected ; HE = High Explosive (conventional shell); N/A = not analyzed; DBS: Donovan Blast Sheet; RDX: RDX blast sheet

**Table 9.19. Phase II. Destruction of DA Type 77 MM Shells. Residual DA and As in Expansion Chamber (EC), Duct (D), on the Walls of the Detonation Chamber (DC) and in Pea Gravel. All numbered shells contained DA.**

Order of detonation	Date	Type	Idf number	Donor Explosive and Additives	Residual DA					As	
					EC (mg/m <sup>3</sup> )	Exhaust Duct (mg/m <sup>3</sup> )	Wall DC (mg/m <sup>2</sup> )	Pea gravel (mg/kg)	DC wall (mg/m <sup>2</sup> )	Pea gravel (mg/kg)	
18	12June	DA	3165	RDX, 2 lbs; 4 bags H2O	ND	0,11	161	13	N/A	550	
19	12June	DA	3152	DBS, 2 lbs; 4 bags of H2O	0,042	0,14	75	183	N/A	1450	
20	12June	DA	3199	RDX, 2 lbs; 4 bags H2O	ND	0,21	116	106	N/A	625	
21	12June	DA	3196	DBS, 2 lbs; 4 bags of H2O	ND	0,06	205	135	N/A	1129	
22	13June	DA	3193	DBS, 2 lbs; 2 bags of H2O; 2bags of Al2O3	0,091	0,07	138	403	N/A	826	
23	13June	DA	3194	DBS, 2 lbs; 2 bags of H2O; 2 bags of Al2O3	0,040	0,05	484	99	N/A	517	
24	13June	DA	3183	DBS 2 lbs, 4 bags of H2O; 2 bags of Al2O3	0,113	0,02	203	166	N/A	1573	
25	13June	DA	3184	DBS, 2 lbs; 4 bags of H2O; 2 bags of Al2O3	0,004	0,16	164	379	N/A	402	
26	14June	DA	3185	DBS, 2 lbs; 4 bags of H2O; 2 bags of Kaolinite	0,116	0,08	276	1123	116	1163	
27	14June	DA	3176	DBS, 2 lbs; 4 bags of H2O; 2 bags of Kaolinite	0,048	0,35	1261	1258	44	1257	
28	14June	DA	3182	DBS, 2 lbs; 4 bags of H2O; 2 bags of Silica gel	0,048	0,32	69	1095	53	1369	
29	14June	DA	3164	DBS, 2 lbs; 2 bags of H2O; 2 bags of H2O2; 2 bags of Al2O3	0,083	0,37	255	803	80	1664	
30-40	15June	DA	3172; 3221; 3220; 3212; 3223; 3205; 3201; 3206; 3208; 3219; 3243; 3242; 3263; 3237;	DBS, 2 lbs; 4 bags of H2O; DBS, 2 lbs; 4 bags of H2O; DBS(+RDX), 4 bags of H2O; BX-24, 1 kg; RDX	N/A	N/A	N/A	866	N/A	893	
41-44	15June	DA	3235	DBS, 2 lbs; 4 bags of H2O; DBS(+RDX), 4 bags of H2O; BX-24, 1 kg; RDX	N/A	N/A	133	1235	115	805	
81	5July	DA	3318	DBS(+RDX), 4 bags of H2O; BX-24, 1 kg; RDX	6,847	5,29	ND	31	98	335	
82	5July	HE	N/A	RDX	N/A	N/A	N/A	N/A	74	304	
83	5July	DA	3317	DBS(+RDX), 4 bags of H2O; BX-24, 1 kg; RDX	10,135	4,93	88	151	37	537	
84	5July	HE	N/A	RDX	N/A	N/A	N/A	N/A	23	526	
85-89	9July	DA	3229; 3232; 3239; 3240; N/A	DBS(+RDX), 4 bags of H2O; RDX	18,175	N/A	9	560	187	1664	
90	9July	HE	N/A	RDX	13,394	13,44	N/A	411	24	646	

**Table 9.20. Phase II. Destruction of DA Type 77 MM Shells. Residual Metal Concentration on the Walls of the Detonation Chamber (DC) and in Pea Gravel.**

					Heavy Metals					
					Al		Cu		Pb	
Order of detonation	Date	Type	Idf number	Donor Explosive and Additives	DC wall (mg/m <sup>2</sup> )	Pea gravel (mg/kg)	DC wall (mg/m <sup>2</sup> )	Pea gravel (mg/kg)	DC wall (mg/m <sup>2</sup> )	Pea gravel (mg/kg)
18	12June	DA	3165	RDX, 2 lbs; 4 bags H2O	N/A	4107	N/A	413	N/A	48
19	12June	DA	3152	DBS, 2 lbs; 4 bags of H2O	N/A	8734	N/A	1582	N/A	190
20	12June	DA	3199	RDX, 2 lbs; 4 bags H2O	N/A	4823	N/A	1238	N/A	236
21	12June	DA	3196	DBS, 2 lbs; 4 bags of H2O	N/A	9326	N/A	2200	N/A	436
22	13June	DA	3193	DBS, 2 lbs; 2 bags of H2O; 2bags of Al2O3	N/A	8091	N/A	1771	N/A	432
23	13June	DA	3194	DBS, 2 lbs; 2 bags of H2O; 2 bags of Al2O3	N/A	7256	N/A	1195	N/A	234
24	13June	DA	3183	DBS 2 lbs, 4 bags of H2O; 2 bags of Al2O3	N/A	16393	N/A	1812	N/A	313
25	13June	DA	3184	DBS, 2 lbs; 4 bags of H2O; 2 bags of Al2O3	N/A	6603	N/A	5410	N/A	950
26	14June	DA	3185	DBS, 2 lbs; 4 bags of H2O; 2 bags of Kaolinite	520	11006	49	1481	10	361
27	14June	DA	3176	DBS, 2 lbs; 4 bags of H2O; 2 bags of Kaolinite	118	14693	16	2197	4	454
28	14June	DA	3182	DBS, 2 lbs; 4 bags of H2O; 2 bags of Silica Gel	235	15333	16	3323	4	438
29	14June	DA	3164	DBS, 2 lbs; 2 bags of H2O; 2 bags of H2O2; 2 bags of Al2O3	631	17312	12	3364	2	468
30-40	15June	DA	3172; 3221; 3220; 3212; 3223; 3205; 3201; 3206; 3208; 3219; 3243;	DBS, 2 lbs; 4 bags of H2O; DBS, 2 lbs; 4 bags of H2O; DBS(+RDX), 4 bags of H2O; BX-24, 1 kg;	N/A	6304	N/A	2124	N/A	406
41-44	15June	DA	3242; 3263; 3237; 3235	DBS, 2 lbs; 4 bags of H2O; DBS(+RDX), 4 bags of H2O; BX-24, 1 kg;	520	6662	49	804	13	142
81	5July	DA	3318	RDX	1011	20345	17	535	2	92
82	5July	HE	NA	RDX	2628	21445	32	664	8	141
83	5July	DA	3317	DBS(+RDX), 4 bags of H2O; BX-24, 1 kg;	317	18202	12	290	2	0
84	5July	HE	NA	RDX	474	19206	8	747	2	148
85-89	9July	DA	3229; 3232; 3239; 3240; Not Registered	DBS(+RDX), 4 bags of H2O; RDX	89	17312	12	3364	2	468
90	9July	HE	NA	RDX	743	10407	41	2559	7	552



**Table 9.21. Phase II. Destruction of DA Type 77 MM Shells.  
Volatile Aromatic Hydrocarbons (VAC)  
in Expansion Chamber (EC).**

Order of detonation	Date	Type	Idf number	Donor Explosive and Additives	VAC (mg/m <sup>3</sup> ) in EC		
					Benzene	Toluene	Xylenes
18	12-Jun	DA	3165	RDX, 2 lbs; 4 bags H <sub>2</sub> O	177.0	2.8	0.2
19	12-Jun	DA	3152	DBS, 2 lbs; 4 bags of H <sub>2</sub> O	22.6	3.6	0.1
20	12-Jun	DA	3199	RDX, 2 lbs; 4 bags H <sub>2</sub> O	23.3	3.1	0.2
21	12-Jun	DA	3196	DBS, 2 lbs; 4 bags of H <sub>2</sub> O	92.4	1.2	0.0
22	13-Jun	DA	3193	DBS, 2 lbs; 2 bags of H <sub>2</sub> O; 2 bags of Al <sub>2</sub> O <sub>3</sub>	82.2	0.4	0.2
23	13-Jun	DA	3194	DBS, 2 lbs; 2 bags of H <sub>2</sub> O; 2 bags of Al <sub>2</sub> O <sub>3</sub>	23.5	2.3	0.1
24	13-Jun	DA	3183	DBS 2 lbs, 4 bags of H <sub>2</sub> O; 2 bags of Al <sub>2</sub> O <sub>3</sub>	21.8	4.0	0.2
25	13-Jun	DA	3184	DBS, 2 lbs; 4 bags of H <sub>2</sub> O; 2 bags of Al <sub>2</sub> O <sub>3</sub>	32.7	2.6	0.1
26	14-Jun	DA	3185	DBS, 2 lbs; 4 bags of H <sub>2</sub> O; 2 bags of Kaolinite	27.9	ND	ND
27	14-Jun	DA	3176	DBS, 2 lbs; 4 bags of H <sub>2</sub> O; 2 bags of Kaolinite	42.9	0.2	ND
28	14-Jun	DA	3182	DBS, 2 lbs; 4 bags of H <sub>2</sub> O; 2 bags of Silica gel	ND	ND	ND
29	14-Jun	DA	3164	DBS, 2 lbs; 2 bags of H <sub>2</sub> O; 2 bags of H <sub>2</sub> O <sub>2</sub> ; 2 bags of Al <sub>2</sub> O <sub>3</sub>	50.5	ND	ND
30-40	15-Jun	DA	3172; 3221; 3220; 3212; 3223; 3205; 3201; 3206; 3208; 3219; 3243;	DBS, 2 lbs; 4 bags of H <sub>2</sub> O;	N/A	N/A	N/A

**Table 9.22. Destruction of DA Type 77 MM Shells.  
Volatile Aromatic Hydrocarbons (VAC) in Expansion Chamber (EC).  
(Continued)**

Order of detonation	Date	Type	Idf number	Donor Explosive and Additives	VAC (mg/m <sup>3</sup> ) in EC		
					Benzene	Toluene	Xylenes
41-44	15 June	DA	3242; 3263; 3237; 3235	DBS, 2 lbs; 4 bags of H <sub>2</sub> O;	N/A	N/A	N/A
81	5 July	DA	3318	DBS(+RDX), 4 bags of H <sub>2</sub> O; BX-24, 1 kg;	1.1	ND	ND
82	5 July	HE	N/A	RDX	N/A	N/A	N/A
83	5 July	DA	3317	DBS(+RDX), 4 bags of H <sub>2</sub> O; BX-24, 1 kg;	108.4	3.3	1.2
84	5 July	HE	N/A	RDX	N/A	N/A	N/A
85-89	9 July	DA	3229; 3232; 3239; 3240; N/A	DBS(+RDX), 4 bags of H <sub>2</sub> O;	N/A	N/A	N/A
90	9 July	HE	N/A	RDX	N/A	N/A	N/A

**Table 9.23. Phase II. Residual Mustard in Expansion Chamber (EC),  
Duct and Detonation Chamber (DC)**

Order of detonation	Date	Shell	Donor Explosive and Additives	Residual HD				
				EC (mg/m <sup>3</sup> )	EC (mg/m <sup>3</sup> ) <sup>1</sup>	Duct (mg/m <sup>3</sup> )	Wall DC (mg/m <sup>2</sup> )	Pea Gravel (mg/kg)
45	20 June	2665	DBS, 5 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	ND	>	1.23	1.2
46	20 June	2788	DBS, 5 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	0.16	0.22	3.18	30.5
47	20 June	2659	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>4</sub>	N/A	0.49	0.43	2.74	10.4
48	20 June	2662	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>5</sub>	ND	0.34	0.17	6.54	19.3
49	21 June	2780	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>6</sub>	N/A	ND	0.15	54.20	5.1
50	21 June	2807	DBS, 6 bags H <sub>2</sub> O	N/A	ND	0.09	76.46	30.9
51	21 June	3103	DBS, 8 bags H <sub>2</sub> O	N/A	ND	0.59	77.10	5.2
52	21 June	3104	DBS, 10 bags H <sub>2</sub> O	N/A	ND	0.88	78.65	31.0
53	22 June	3106	DBS, 10 bags H <sub>2</sub> O	1.81	9.09	0.36	46.67	60.7
54	22 June	3135	DBS, 8 bags H <sub>2</sub> O, 2 bags H <sub>2</sub> O <sub>2</sub>	2.37	3.02	0.55	6.92	7.0
74	3 July	3043	DBS, 4 bags of H <sub>2</sub> O, 2 bags of Al <sub>2</sub> O <sub>3</sub>	N/A	ND	ND	11.31	6.6
75	3 July	HE	DBS, 4 bags of H <sub>2</sub> O,	N/A	0.28	0.59	ND	39.3
76	3 July	3033	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>4</sub>	N/A	0.11	ND	0.39	6.9
77	3 July	HE	DBS, 4 bags H <sub>2</sub> O	N/A	0.25	0.24	1.28	64.4
78	4 July	3132	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	N/A	ND	ND	4.40	4.4
79	4 July	3056	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	ND	ND	0.93	1.33	56.0

**Table 9.24. Phase II. Residual Chlorobenzene (CB) in Expansion Chamber (EC),  
Detonation Chamber (DC) and Duct**

Order of detonation	Date	Shell	Donor Explosive and Additives	Residual CB				
				EC (µg/cm)	EC (µg/cm)	Duct (µg/cm)	Wall DC (mg/sqm)	Pea gravel (mg/kg)
45	20 June	2665	DBS, 5 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	725, 5	7412	>	1.5	1.2
46	20 June	2788	DBS, 5 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	1601	2251	3836	2.0	2.3
47	20 June	2659	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>4</sub>	N/A	30736	280	13.0	1.8
48	20 June	2662	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>5</sub>	4561	4806	146	1.1	1.3
49	21 June	2780	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>6</sub>	N/A	23723	315	0.0	7.5
50	21 June	2807	DBS, 6 bags H <sub>2</sub> O	N/A	18530	28	47.3	8.7
51	21 June	3103	DBS, 8 bags H <sub>2</sub> O	N/A	2865	1050	1.8	10.1
52	21 June	3104	DBS, 10 bags H <sub>2</sub> O	N/A	>	435	14.4	9.0
53	22 June	3106	DBS, 10 bags H <sub>2</sub> O	56917	15254	6662	10.9	12.0
54	22 June	3135	DBS, 8 bags H <sub>2</sub> O, 2 bags H <sub>2</sub> O <sub>2</sub>	75747	6292	7146	10.0	4.9
74	3 July	3043	DBS, 4 bags of H <sub>2</sub> O, 2 bags of Al <sub>2</sub> O <sub>3</sub>	N/A	789	730644	53.8	36.7
75	3 July	HE	DBS, 4 bags of H <sub>2</sub> O,	N/A	1465	17752	48.1	10.7
76	3 July	3033	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>4</sub>	N/A	844	268223	37.0	2.2
77	3 July	HE	DBS, 4 bags H <sub>2</sub> O	N/A	2207	1532	5.0	49.2
78	4 July	3132	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	N/A	609	60204	50.9	6.0
79	4 July	3056	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	N/A	1914	16337	50.9	41.7

<sup>1</sup>In some cases two samples were taken from the EC

**Table 9.25. Phase II. Volatile Aromatic Hydrocarbons (VAC) Concentrations in Expansion Chamber (EC) for Detonations of HD Shells**

Order of detonation	Date	Shell	Donor Explosive and Additives	VAC		
				Benzene	Toluene	Xylene
45	20 June	2665	DBS, 5 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	N/A	N/A	N/A
46	20 June	2788	DBS, 5 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	N/A	N/A	N/A
47	20 June	2659	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>4</sub>	N/A	N/A	N/A
48	20 June	2662	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>5</sub>	N/A	N/A	N/A
49	21 June	2780	DBS, 6 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>6</sub>	N/A	N/A	N/A
50	21 June	2807	DBS, 6 bags H <sub>2</sub> O	9.0	ND	1.1
51	21 June	3103	DBS, 8 bags H <sub>2</sub> O	9.5	ND	ND
52	21 June	3104	DBS, 10 bags H <sub>2</sub> O	7.2	ND	0.8
53	22 June	3106	DBS, 10 bags H <sub>2</sub> O	3.3	ND	0.5
54	22 June	3135	DBS, 8 bags H <sub>2</sub> O, 2 bags H <sub>2</sub> O <sub>2</sub>	N/A	N/A	N/A
74	3 July	3043	DBS, 4 bags of H <sub>2</sub> O, 2 bags of Al <sub>2</sub> O <sub>3</sub>	23.7	0.6	ND
75	3 July	HE	DBS, 4 bags of H <sub>2</sub> O,	16.0	2.3	0.1
76	3 July	3033	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>4</sub>	22.8	0.6	ND
77	3 July	HE	DBS, 4 bags H <sub>2</sub> O	11.8	5.8	ND
78	4 July	3132	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	29.2	223.0	0.2
79	4 July	3056	DBS, 4 bags H <sub>2</sub> O, 2 bags Al <sub>2</sub> O <sub>3</sub>	47.7	6.1	0.2

**Table 9.26. Phase II. Residues of Elemental Arsenic (As), Aluminum (Al), Copper (Cu), and Lead (Pb) in Expansion Chamber (EC) (Airborne) and Pea Gravel for Detonation of HD Shells**

				Heavy Metals							
Order of detonation	Date	Shell	Donor Explosive and Additives	As		Al		Cu		Pb	
				As airborne EC (µg/cum)	Pea gravel (mg/kg)	Al airborne EC (µg/cum)	Pea gravel (mg/kg)	Cu airborne EC (µg/cum)	Pea gravel (mg/kg)	Pb airborne EC (µg/cum)	Pea gravel (mg/kg)
45	20 June	2885	DBS, 5 bags H <sub>2</sub> O, 2 bags Al2O3	8.09	83	11.5	14287	2.52	936	1.31	171
46	20 June	2788	DBS, 5 bags H <sub>2</sub> O, 2 bags Al2O3	5.05	ND	16.2	12700	1.63	689	ND	ND
47	20 June	2859	DBS, 4 bags H <sub>2</sub> O, 2 bags Al2O4	1.45	ND	3.82	10619	0.54	831	ND	179
48	20 June	2882	DBS, 4 bags H <sub>2</sub> O, 2 bags Al2O5	ND	ND	1.49	12359	0.21	1135	ND	224
49	21 June	2780	DBS, 6 bags H <sub>2</sub> O, 2 bags Al2O8	0.05	ND	2.34	12553	0.37	1868	ND	404
50	21 June	2807	DBS, 6 bags H <sub>2</sub> O	ND	ND	2.92	14207	0.56	1030	ND	170
51	21 June	3103	DBS, 8 bags H <sub>2</sub> O	ND	ND	4.65	13045	0.5	1835	ND	438
52	21 June	3104	DBS, 10 bags H <sub>2</sub> O	ND	ND	3.78	10086	0.47	2148	ND	500
53	22 June	3108	DBS, 10 bags H <sub>2</sub> O	ND	ND	4.41	12488	0.57	1152	ND	353.5
54	22 June	3135	DBS, 8 bags H <sub>2</sub> O, 2 bags H2O2	ND	ND	4.31	16139	0.56	1046	ND	153.8
75	3 July	3043	DBS, 4 bags of H <sub>2</sub> O, 2 bags of Al2O3	ND	ND	5.54	22127	1.39	554		86
76	3 July	HE	DBS, 4 bags of H <sub>2</sub> O	ND	ND		21541		1780		395
77	3 July	3033	DBS, 4 bags H <sub>2</sub> O, 2 bags Al2O4	ND	ND	4.19	21954	0.67	1503	0.93	322
78	3 July	HE	DBS, 4 bags H <sub>2</sub> O	ND	ND	10.43	23780	0.33	662	ND	119
79	4 July	3132	DBS, 4 bags H <sub>2</sub> O, 2 bags Al2O3	ND	ND	17.23	13422	2.55	2160	ND	452
	4 July	3056	DBS, 4 bags H <sub>2</sub> O, 2 bags Al2O3	ND	ND	7.99	16251	1.63	1148	ND	244

**Table 9.27. Phase II. Airborne Concentrations of CWA Measured at the Exhaust Vent of the Carbon/HEPA Filtration System.**  
Values are in Mg/m<sup>3</sup>

Date (2001)	6/15	6/20	6/21	6/22	6/26	6/27	6/28	6/29	7/3	7/4	7/5	7/8	7/11	7/12
HD	ND	0.0078	0.0452	0.0927	ND	ND	ND	ND	ND	0.0076	0.0102	0.0018	0.0031	0.1097
CB	ND	0.00154	0.00240	0.05675	ND	ND	ND	ND	ND	0.00672	0.06572	0.00083	0.00291	0.26369
DA	0.27	0.22	ND	ND	ND	ND	ND	ND	ND	ND	1.34	0.05	0.17	ND
PS	ND	ND	ND	ND	0.4248	0.5837	1.2210	2.8649	ND	ND	ND	ND	ND	
CG	ND	ND	ND	ND	0.0261	0.0307	ND	0.0839	ND	ND	ND	ND	ND	

Remark: ND = not detected;  
Detection Limits (in mg/m<sup>3</sup>) are 0.0001 for HD and Chloropicrin, 0.0002 for Phosgene, 0.000002 for Chlorobenzene and 0.013 for DA

**Table 9.28. Phase II. Airborne Concentrations of CWA in mg/m<sup>3</sup> from Personal Sample Monitoring**

Date (2001)	DA	HD	CB	CG	PS	Remarks
6/12	0.55	ND	ND	ND	ND	4 shells DA detonated
6/13	0.27	ND	ND	ND	ND	4 shells DA detonated
6/14	0.14	ND	ND	ND	ND	4 shells DA detonated
6/15	183	ND	ND	ND	ND	15 shells DA detonated
6/19	148	ND	ND	ND	ND	Decon post DA
6/20	ND	0.038	0.013	ND	ND	4 shells HD detonated
6/21	ND	0.734	0.229	ND	ND	4 shells HD detonated
6/22	ND	0.028	0.040	ND	ND	PVC, TNT, 2 shells HD
6/25	ND	ND	0.103	ND	ND	Demo Detonation
6/26	ND	ND	0.053	ND	ND	Decon post HD
6/26	ND	0.429	0.085	ND	ND	Decon post HD
6/26	ND	0.037	0.028	ND	ND	Decon post HD
6/29	ND	ND	ND	ND	0.194	5 Shells CG detonated
7/2	ND	0.004	0.0005	ND	ND	Decon post CG
7/3	ND	0.047	0.011	ND	ND	2 HE shells + 2 HD shells detonated
7/4	ND	0.042	0.228	ND	ND	2 HD shells detonated
7/5	ND	0.005	0.005	ND	ND	1 CG shell, 2 HE shells + 2 DA shells detonated
7/9	ND	ND	0.0004	ND	ND	Decon

Remark: ND = not detected;  
Detection Limits (in mg/m<sup>3</sup>) are 0.000001 for HD and Chloropicrin, 0.0002 for Phosgene, 0.000002 for Chlorobenzene, and 0.00002 for DA

**Table 9.29. Phase II. Residues in Expansion Chamber (EC) and Detonation Chamber (DC).**

Values are in mg/kg solid ash/soot

	Floor (EC)			Door (EC)			Wall (DC)			Pea Gravel (DC)		
	6/18	6/25	7/9	6/18	6/25	7/9	6/18	6/25	7/9	6/18	6/25	7/9
Date (2001)	14621	19463	18457	25309	27068	21107	15777	N/A	13203	5986	9242	13043
As	4293	4079	3997	13125	1589	2760	7198	N/A	2216	625	ND	1154
Cu	7363	1854	1448	2113	1231	897	957	N/A	491	204	695	1134
Pb	1670	490	415	786	153	226	409	N/A	140	44	123	198
DA	7138	3228	ND	10749	522	ND	ND	N/A	ND	113	ND	560
HD	ND	3.61	20.18	ND	116.08	7.6	ND	15.45	7.46	ND	11.43	4.87
CB	ND	3.84	32.97	ND	3.18	12.8	ND	4.33	14.1	ND	1.3	5.14
CG	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
PS	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
DIP	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

**Table 9.30. Residual Quantities on Bag House Filters (g/filter)**

	Filter posn 1		Filter posn 1		Filter posn 2	
	Date Sampled (2001)		6/19		7/09	
Agent						
CB			1.52	25.98	0.82	0.61
H			2.42	0.74	0.29	0.12
DA			31.62	ND	ND	ND
AI			115.83	38.32	59.00	124.38
As			37.04	27.11	32.09	69.49
Cu			11.34	4.62	7.63	13.32
Pb			4.10	1.41	2.35	4.81

**Table 9.31. Resulting Waste Stream From the Two Test Phases**

Description	Weight (Tons)
Decontamination liquid (water + Hypochlorite)	0.8
Pea Gravel (including iron scrap)	1.9
Protective equipment and cleaning towels	0.3
HEPA filters	0.2
Total	3.2

**Table 9.32. Estimation of Undestroyed Mass of CWA (grams/shell)**

	Expansion Chamber (airborne)	Duct	Wall DC	Pea gravel	Ashes/soot in Expansion Chamber	NORIT Filter	Exhaust Vent	Total Mass Undestroyed
DA	0.0004	0.0003	3.27	23.00	13.96	7.02	0.17	47.41
HD	0.0124	0.0008	0.42	1.50	0.02	0.44	0.04	2.45
CB	117.3	4.42	0.12	2.06	ND	ND	0.01	123.95
PS	0.061	0.063	0.012	ND	ND	ND	1.404	1.54
CG	0.006	0.007	ND	ND	ND	ND	0.035	0.05



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# STANDARD B

## PREFACE

The work described in this report was sponsored by the Office of the Deputy Assistant Secretary of the Army for the Environment, Safety, and Occupational Health and the Belgian Ministry of Defense (MOD). This work was started in May 2001 and completed in July 2001.

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## Acknowledgments

The authors thank John L. Schwarz, Chemist, U.S. Army Edgewood Chemical Biological Center (ECBC) and Sr. Captain Bart Smedts, Chemist, Royal Military Academy (RMA) of Belgium, whose significant contributions helped complete this evaluation.

The authors also recognize M.H. Lefebvre, Professor, Applied Chemistry, RMA; Christopher C. Keiser, Engineer, ECBC; J.L. Donovan, Chairman, DeMil International; P.J.J. Lodewyckx, Material Resources, Belgian Armed Forces; and W.J. Mitchell, Scientist, W.J. Mitchell and Associates, for their contributions to the evaluation.

The authors also wish to thank the operators of the Contained Detonation Chamber, both of Demil, International, and the Belgium EOD Bomb Disposal Unit (DOVO), Poelkapelle, for their efforts. In addition, the authors thank the scientists at RMA, DOVO, and ECBC for their efforts in collection, analysis, and interpretation of the data. Finally, a special thank you to all U.S and Belgian Officials who contributed to the realization of this project.

This project was made possible by grant F00/16 of the Belgian MOD and funding from the Office of the Deputy Assistant Secretary of the Army for the Environment, Safety, and Occupational Health.

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